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FOREWORD

The *Proceedings* of the American Society for Testing Materials are published annually and include all reports and papers offered to the Society during the current year and accepted by the Administrative Committee on Papers and Publications for the *Proceedings*, together with discussion.

The table of contents and subject and author indexes cover all papers and reports published by the Society during the current year, which in addition to those appearing in the *Proceedings* include those accepted for publication in the ASTM BULLETIN or in Special Technical Publications.

A list of the Special Technical Publications published by the Society in 1958 is given on page 1420 of this volume. This supplements the lists appearing in the *Proceedings* from 1948 to 1957 covering all special publications published by the Society up to and including 1957.

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AMERICAN SOCIETY FOR TESTING MATERIALS

1916 RACE ST., PHILADELPHIA 3, PA.

SUMMARY OF PROCEEDINGS

OF THE

SIXTY-FIRST ANNUAL MEETING

BOSTON, MASS., JUNE 22–27, 1958

This summary of the Sixty-first Annual Meeting of the American Society for Testing Materials, held at the Statler and Sheraton-Plaza Hotels, Boston, Mass., June 22–27, 1958, is a record of the transactions of the meeting, including the actions taken on the various recommendations submitted by the technical committees. In all, 42 technical sessions were held.

The registered attendance of the meeting is as follows: Members present or represented, 1802; committee members, 722; guests, 471; total, 2995; ladies, 500.

The Proceedings are set forth session by session. There were 72 reports and 144 formal papers presented. The record with respect to each has been briefed; the recommendations in the reports have been grouped so as to cover the acceptance of material for publication as tentative, such as new specifications, methods of test, revisions of tentatives, and proposed revisions of existing standards, and, as a separate group, the approval of matters that were referred to letter ballot of the Society, comprising the adoption of tentatives as standard and the adoption of revisions of standards. Accordingly, wherever the action is reported "adopted as standard" or "adopted as standard, revisions in," it is understood that this indicates approval of the Annual Meeting for reference to letter ballot of the Society.[1] The various recommendations so recorded are included in the Society letter ballot. The actions designated as "accepted as tentative" or "accepted as tentative, revisions in" are self-evident as indicating acceptance by the Society at the Annual Meeting for publication as tentative. Designations that have since been assigned to new tentatives are included as information.

All papers, unless otherwise noted, are published in the *Proceedings*.

While all the items on the program are recorded under the particular session in which they are presented, for convenience in locating actions with respect to any particular report, the accompanying list is presented of all reports together with the page reference where the actions thereon are recorded. There are also listed the various symposia and special sessions.

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[1] The letter ballot on recommendations affecting standards, distributed to the Society membership, will be canvassed on September 22, 1958. See Editor's Note, p. 42.

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FIRST SESSION—OPENING SESSION

MONDAY, JUNE 23, 10:00 A.M.

FORMAL OPENING OF THE SIXTY-FIRST ANNUAL MEETING, PRESIDENT R. T. KROPF

The session was formally opened by President R. T. Kropf, who called attention to the very complete program of events that were to follow during the ensuing week. The meeting was then turned over to the session chairman, D. G. Richards, Hamilton Standard Division of United Aircraft Corp.

SESSION ON FATIGUE

SESSION CHAIRMAN: D. G. RICHARDS

Tentative Fatigue Strength Reduction Factors for Silicate-Type Inclusions in High Strength Steels—H. N. Cummings, F. B. Stulen, and W. C. Schulte, presented by Mr. Stulen.

Torsional Fatigue Properties of Small Diameter High-Carbon Steel Wire—H. C. Burnett, presented by the author.

Effects of Grinding Direction and of Abrasive Tumbling on the Fatigue Limit of Hardened Steel—L. P. Tarasov, W. S. Hyler, and H. R. Letner, presented by Mr. Tarasov.

Distribution of Fatigue Failures in Flat Hardened Steel Test Bars—L. P. Tarasov, W. S. Hyler, and R. J. Favor, presented by Mr. Hyler.

Effect of Static Prestrain on the Fatigue Properties—F. Vitovec, presented by the author.

(*Continued in Tenth Session*)

SYMPOSIUM ON INSTRUMENTATION

MONDAY, JUNE 23, 10:00 A.M.

SESSION CHAIRMAN: M. D. THOMAS

Subcommittee IV on Instrumentation of Committee D-22 on Methods of Atmospheric Sampling and Analysis sponsored a Symposium on Instrumentation at which the following papers were presented:

Light Scattering Instrumentation for Characterization of Aerosols—Chester T. O'Konski.

Filtration Methods for Evaluation of Aerosol Contaminants—Morris Katz and H. P. Sanderson.

Relative Merits of Gas Chromatography, Colorimetry and Spectrometry for Air Pollution Measurements—Lewis A. Rogers.

Discussion of Atmospheric Fluoride Recorder—Moyer Thomas and D. A. St. John.

SECOND SESSION—SYMPOSIUM ON BASIC MECHANISMS OF FATIGUE[2]

MONDAY, JUNE 23, 2:00 P.M.

SESSION CHAIRMAN: T. J. DOLAN

Dislocation Behavior in Lithium Fluoride Crystals During Cyclic Stressing—R. E. Keith and J. J. Gilman, presented by Mr. Keith.

Cycle-Dependent Stress Relaxation—JoDean Morrow and G. M. Sinclair, presented by Mr. Morrow.

Internal Friction, Plastic Strain, and Fatigue in Metals and Semiconductors—W. P. Mason, presented by the author.

Observations Relating to a Proposed Mechanism of Fracture by Fatigue—W. A. Wood, presented by H. T. Corten.

A Study of Fatigue Crack Formation in Silver Chloride—P. J. E. Forsyth, presented by W. S. Hyler.

Slipband Formation and Fatigue Cracks of Different Materials Under Alternating Stressing—Max R. Hempel, presented by D. G. Richards.

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[2] To be issued as separate publication *ASTM STP No. 237.*

THIRD SESSION—SYMPOSIUM ON SOME APPROACHES TO DURABILITY IN STRUCTURES[3]

MONDAY, JUNE 23, 2:00 P.M.

SESSION CHAIRMAN: E. C. SHUMAN

Some Factors Affecting Durability of Structural Clay Product Masonry—P. V. Johnson and H. C. Plummer, presented by Mr. Johnson.

Laboratory Testing and the Durability of Concrete—T. B. Kennedy, presented by the author.

Durability Tests of Structural Sandwich—E. W. Kuenzi and L. W. Wood, presented by Mr. Kuenzi.

The Durability of Buildings—R. F. Legget and N. B. Hutcheon, presented by Mr. Legget.

Effect of the Atmosphere on Masonry and Related Materials—J. W. McBurney, presented by the author.

Relation Between Actual and Artificial Weathering of Organic Materials—F. W. Reinhart, presented by the author.

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[3] To be issued as separate publication *ASTM STP No. 236.*

FOURTH SESSION—SYMPOSIUM ON SOLVENT EXTRACTION IN THE ANALYSIS OF METALS[4]

MONDAY, JUNE 23, 2:00 P.M.

SESSION CHAIRMAN: GEORGE H. MORRISON

Convergence of Tie Lines in Ternary Liquid Systems—Application to Liquid Extraction—R. L. Pilloton, presented by the author.

Metals Analysis with Thenoyltrifluoroacetone—F. L. Moore, presented by the author.

The Use of Tri-*n*-Octylphosphine Oxide in Analytical Chemistry—J. C. White, presented by the author.

8-Hydroxyquinaldine Extractions Applied to the Analysis of Metals—R. J. Hynek, presented by the author.

The Role of Organic Solvents in Flame Photometry—J. A. Dean, presented by the author.

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[4] To be issued as separate publication *ASTM STP No. 238.*

FIFTH SESSION—SYMPOSIUM ON BULK SAMPLING[5]

In cooperation with the Chemical Division of the American Society for Quality Control

MONDAY, JUNE 23, 2:00 P.M.

SESSION CHAIRMAN: SIMON COLLIER

Measurement Error Considerations in Bulk Sampling—A. J. Duncan, presented by the author.

Prepared Discussion, Edward Forst, Jr.

An Interpretation of the Enos Coal Sampling Data as an Example of Bulk Sampling—B. A. Landry, presented by the author.

Prepared Discussion, G. W. Armstrong.

The Effect of Increment Weight on Sampling Accuracy—W. M. Bertholf, presented by the author.

Prepared Discussion, Alonzo Church, Jr.

MONDAY, JUNE 23, 8:00 P.M.

SESSION CHAIRMAN: B. H. LLOYD

The Purposes of Sampling—W. E. Deming, presented by the author.

Studies in Ore Car and Abrasive Grain Sampling Variation—R. S. Bingham, Jr., J. L. Gioele, and V. B. Shelburne, presented by Mr. Gioele.

Prepared Discussion, N. F. Knowlden.

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[5] To be issued as separate publication *ASTM STP No. 242.*

SIXTH SESSION—COMMITTEE REPORT SESSION

MONDAY, JUNE 23, 4:30 P.M.

SESSION CHAIRMAN: M. A. PINNEY

Committee C-2 on Magnesium Oxychloride and Magnesium Oxysulfate Cements:

Report presented in the absence of the chairman by N. V. Becker, and the following actions taken:

Adopted as Standard:

Def. of Terms Relating to Magnesium Oxychloride and Magnesium Oxysulfate Cements (C 376 - 55 T)

Test for Yield of Magnesium Oxychloride Cement (Field Test) (C 388 - 56 T)

Test for Yield of Magnesium Oxychloride Cement (Laboratory Test) (C 389 - 56 T)

Committee D-7 on Wood:

Report presented in the absence of the chairman by S. F. Etris, and the following actions taken:

Accepted as Tentative:

Def. of Terms Relating to Wood-Base Fiber and Particle Panel Materials (D 1554 - 58 T)

Accepted as Tentative, Revisions in:

Spec. for Wood Paving Blocks for Exposed Platforms, Pavements, Driveways, and Interior Floor Exposed to Wet and Dry Conditions (D 52 - 57) (Standard reverted to tentative)

Adopted as Standard:

Methods of Static Tests of Wood Poles (D 1036 - 55 T)

Adopted as Standard, Revisions in:

Spec. for Round Timber Piles (D 25 - 55)

Test for Water in Creosote (D 370 - 33)

Test for Integrity of Glue Joints in Laminated Wood Products for Exterior Service (D 1101 - 53)

Committee E-5 on Fire Tests of Materials and Construction:

Report presented in the absence of the chairman by A. L. Brown, and the following actions taken:

Accepted as Tentative:

Test for Defining Noncombustibility of Building Materials (E 136 - 58 T)

Adopted as Standard:

Methods of Fire Tests of Door Assemblies (E 152 – 56 T)
Methods of Fire Tests of Roof Coverings (E 108 – 56 T)

Adopted as Standard, Revisions in:

Methods of Fire Tests of Building Construction and Materials (E 119 – 55): covering (1) adoption as standard of tentative revision of Section 26; and (2) immediate adoption of revisions of Sections 5 and 23.

The committee withdrew from the report as preprinted the recommendation for adoption as standard of Tentative Method of Fire Hazard Classification of Building Materials (E 84 – 50 T).

Committee E-11 on Quality Control of Materials:

Report presented in the absence of the chairman by S. F. Etris, and the following actions taken:

Accepted as Tentative, Revisions in:

Rec. Practices for Designating Significant Places in Specified Limiting Values (E 29 – 50) (Standard reverted to tentative)

Adopted as Standard:

Rec. Practice for Probability Sampling of Materials (E 105 – 56 T)
Rec. Practice for Choice of Sample Size to Estimate the Average Quality of a Lot or Process (E 122 – 56 T)

SEVENTH SESSION—FERROUS METALS

MONDAY, JUNE 23, 8:00 P.M.

SESSION CO-CHAIRMEN: VICE-PRESIDENT F. L. LAQUE
W. T. LANKFORD

Microstructural Observations Pertinent to the Tempering and Temper Brittleness of Steel—W. P. Clancy and Mary R. Norton, presented by Miss Norton.

The Effects of Strain Rate and Temper Rolling on the Strain-Aging Characteristics of Rimmed Deep-Drawing Steel—D. H. Fisher, R. L. Carlson, and W. T. Lankford, presented by Mr. Fisher.

Toughening High Strength Steel by Warm Working—E. J. Ripling, presented by the author.

Effect of Size on the Yielding of Mild Steel Beams—C. W. Richards, presented by the author.

Stress-Rupture and Creep Properties of Malleable Iron at Elevated Temperatures—L. C. Marshall and G. F. Sommer, presented by Mr. Marshall.

Hysteresis and Anelasticity in Cold-Worked Stainless Steel—J. D. Lubahn, presented by the author.

EIGHTH SESSION—CONCRETE

MONDAY, JUNE 23, 8:00 P.M.

SESSION CHAIRMAN: P. H. PETERSEN

Reflectance Tests for Concrete Curing Materials—C. C. Rhodes, M. H. Janson, and M. G. Brown, presented by B. W. Pocock.

Extending Concrete Highway Durability with Silicones—H. L. Cahn and R. V. Mackey, Jr., presented by Mr. Cahn.[6]

Testing of Prestressing Materials and Concrete Control on the Northern Illinois Toll Highway—J. J. Waddell, presented by the author.[7]

Influence of Various Factors on Sulfate Resistance of Concrete—Milos Polivka and E. H. Brown, presented by Mr. Polivka.

(*Continued in Ninth Session*)

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[6] Published in the ASTM BULLETIN, No. 235, Jan., 1959, p. 37 (TP 7).

[7] Published in the ASTM BULLETIN, No. 234, Dec., 1958, p. 35 (TP 247).

SYMPOSIUM ON ADVANCES IN ELECTRON METALLOGRAPHY[8]

TUESDAY, JUNE 24, 9:00 A.M.

Subcommittee XI on Electron Metallography of Metals of Committee E-4 on Metallography held a symposium on advances in electron metallography, with the presentation of the following papers:

First Progress Report of Non-Ferrous Task Group on Metallography of Titanium–8 per cent Manganese Alloy—J. McNutt.

Vibratory Polishing of Specimens for Electron Microscopy—E. L. Long and R. J. Gray.

A Technique for the Easy Removal of "Direct" Replicas for Electron Microscopy—W. H. Bridges and E. L. Long, Jr.

Examination of Metal Foils by Transmission Electron Microscopy—F. W. Boswell.

A Study of Dislocations in Thin Metal Foils Elongated in the Electron Microscope—H. G. F. Wilsdorf.

Microstructure of Age Hardenable Alloys—J. R. Mihalisin and K. G. Carroll.

An Electron Microscopic Study of the Precipitation Hardening of Commercial Nickel-Base Alloys—W. C. Bigelow, C. L. Cory, and J. W. Freeman.

The Structure and Composition of the γ' Phase of Commercial Nickel Base Alloys—J. A. Amy and W. C. Bigelow.

Electron Probe Analysis of Segregation in Inconel—L. S. Birks and E. J. Brooks.

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[8] To be issued as separate publication *ASTM STP No. 245.*

NINTH SESSION—CONCRETE

(Continued from the Eighth Session)

TUESDAY, JUNE 24, 9:30 A.M.

SESSION CHAIRMAN: M. N. CLAIR

Long-Time Creep and Shrinkage Tests of Plain and Reinforced Concrete—G. E. Troxell, J. M. Raphael, and R. E. Davis, presented by Mr. Davis.

The Effect of Fine Aggregate Characteristics Other than Grading on Strength and Durability of Concrete—J. G. Dempsey, presented by the author.

The Effect of Type of Capping Material on the Compressive Strength of Concrete Cylinders—George Werner, presented by the author.

Flexural and Compressive Strength Properties of Air-Entrained Concrete with Blast-Furnace Slag Aggregate—D. W. Lewis and Fred Hubbard, presented by Mr. Lewis.

TENTH SESSION—FATIGUE

(Continued from the First Session)

TUESDAY, JUNE 24, 9:00 A.M.

SESSION CHAIRMAN: JOSEPH MARIN

A Mechanism for Nonpropagating Fatigue Cracks—L. F. Coffin, Jr., presented by the author.

A High Temperature Vacuum and Controlled Environment Fatigue Tester—G. J. Danek, Jr. and M. R. Achter, presented by Mr. Danek.[9]

A Fatigue Hypothesis Based Upon Stabilized Unidirectional Slip—D. G. Younger, presented by the author.

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[9] Published in the ASTM BULLETIN, No. 234, Dec., 1958, p. 48 (TP 260).

ELEVENTH SESSION—SYMPOSIUM ON RADIATION EFFECTS ON MATERIALS[10]

Jointly sponsored with Subcommittee on Radiation Effects of Atomic Industrial Forum

TUESDAY, JUNE 24, 9:30 A.M.

SESSION CO-CHAIRMEN: J. H. KITTEL
A. N. TSCHAECHE

Committee E-10 on Radioisotopes and Radiation Effects:

Report presented by C. E. Crompton, chairman, and accepted as a report of progress.

Symposium Papers:

I. Dosimetry Considerations

Dosimetry Techniques for Gamma and Reactor Radiation Fields—Paul Schall, Jr. and J. F. Kircher, presented by Mr. Kircher.

II. Irradiation Facilities and Techniques

Irradiation Facilities in NRU—G. C. Laurence, presented by the author.

The Engineering Test Reactor as an Irradiation Facility—R. L. Doan, presented by the author.

A Facility for the Study of the Effects of Nuclear Radiation on Materials—J. L. Colp and A. W. Snyder, presented by Mr. Colp.

An In-Pile Fatigue Testing Apparatus—E. E. Drucker, presented by the author.

(Continued in Thirteenth Session)

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[10] To be issued as separate publication *ASTM STP No. 233.*

TWELFTH SESSION—LUNCHEON, PRESIDENT'S ADDRESS, INTRODUCTION OF NEW OFFICERS, RECOGNITION OF 40-YEAR AND 50-YEAR MEMBERS, HONORARY MEMBERSHIPS, REPORT OF BOARD OF DIRECTORS, AWARDS

TUESDAY, JUNE 24, 12:00 NOON

SESSION CHAIRMAN: NATIONAL DIRECTOR A. ALLAN BATES

About 350 were present at this annual luncheon session, including a number of ladies and visitors.

Awards of Merit:

The Chairman of the 1958 Award of Merit Committee, Henry Grinsfelder, read brief citations and presented the following to President Richard T. Kropf, who conferred on them, on behalf of the Board of Directors, the Certificate of Award of Merit:

Oliver P. Beckwith
Theodore Irving Coe
Russell Howard Heilman
Clifford P. Larrabee
* Fritz V. Lenel
James J. Moran
James B. Rather, Jr.
* Arnold Henry Scott
Bourdon Francis Scribner
Myron Allan Swayze
Louis Jacob Trostel
Vincent P. Weaver

* Presented in absentia.

Election of Officers:

Results of the letter ballot on election of new officers were announced by R. A. Pomfret on behalf of the Teller's Committee consisting of Mr. Pomfret, Chairman, and W. L. Glowacki. The results were as follows:

For President, to serve for one year:
  K. B. Woods, 1823 votes.
For Vice-President, to serve for two years:
  A. Allan Bates, 1818 votes.
For Directors, to serve for three years:
  P. A. Archibald, 1815 votes.
  W. L. Fink, 1811 votes.
  H. M. Hancock, 1811 votes.
  L. A. O'Leary, 1813 votes.
  A. C. Webber, 1813 votes.

The newly-elected members of the Board of Directors who were present were introduced, as were President-elect Woods and Vice-President-elect Bates and F. L. LaQue, whose term as Vice-President continues another year.

Award of Honorary Memberships:

The Executive Secretary read citations for the following four members who had been elected by the Board of Directors to Honorary Membership in the Society:

H. J. Ball
T. S. Fuller
J. L. McCloud
J. G. Morrow

Presidential Address:

The annual President's Address was presented by Richard T. Kropf, on the subject "A Continuing and Vital Challenge."

Report of the Board of Directors:

R. J. Painter, Executive Secretary, presented a brief résumé of the Report of the Board of Directors, and submitted for action an amendment to the By-laws increasing the number of Directors from fifteen to eighteen, and increasing the dues of Company Members from $50 to $75 and of Sustaining Members from $150 to $200. These changes affect Article II, Sections 1 and 4, and Article VIII, Section 1. The amendment on motion was approved.

Recognition of 40-Year and 50-Year Members:

Chairman Bates called upon Vice-President K. B. Woods, incoming President, who read the names of the twenty members, both individual and company, who had been continuously affiliated with the Society for 50 years. Certificates to that effect were presented. T. P. Dresser, Jr., of Abbot A. Hanks, Inc., made a brief response on behalf of the 50-Year Members and also invited those present to attend the Third Pacific Area National Meeting to be held in San Francisco the week of October 11, 1959.

Chairman Bates read the names of the 35 members, both individual and company, who had been continuously affiliated with the Society for 40 years, and certificates to that effect were presented.

Special Presentations:

At the close of the session, the Chairman presented the Past-President's pin to retiring President Richard T. Kropf. Mention was made that this meeting brings to a close for N. L. Mochel nine years on the Board of Directors, as Director, Vice-President, President, and Past-President. Recognition was also given to R. C. Alden, E. F. Lundeen, and J. C. Moore as retiring Directors.

THIRTEENTH SESSION—SYMPOSIUM ON RADIATION EFFECTS ON MATERIALS[10]

(Symposium Continued from Eleventh Session)

Jointly sponsored with Subcommittee on Radiation Effects of Atomic Industrial Forum

TUESDAY, JUNE 24, 2:30 P.M.

SESSION CO-CHAIRMEN: J. H. KITTEL
C. R. SUTTON

III. Studies on Radiation Effects

Reactor Pressure Vessel Design for Nuclear Applications—N. Balai, R. E. Bailey, and T. L. Kettles, presented by Mr. Balai.

Fast Neutrons and the EBR-I Core Flow Separator—R. E. Bailey and M. A. Silliman, presented by Mr. Bailey.

Control Materials for Pressurized Water Reactors—W. K. Anderson, D. N. Dunning, and W. E. Ray, presented by Mr. Ray.

Radiation Behavior of Fuel Materials for Sodium Graphite Reactors—B. R. Hayward, L. E. Wilkinson, and C. C. Woolsey, presented by Mr. Woolsey.

Irradiation of Uranium-Fissium Alloys and Related Compositions—K. F. Smith (presented by title only).

The Effects of Nuclear Radiation on Natural Quartz Piezoelectric Crystals—F. E. Graham and A. F. Donovan, presented by Mr. Donovan.

Some Observations on the Effects of High Neutron and Gamma Fluxes on the Transmission Characteristics of Some Optical Glasses—J. L. Colp and H. N. Woodall, presented by Mr. Colp.

FOURTEENTH SESSION—TEXTILES

TUESDAY, JUNE 24, 2:30 P.M.

SESSION CHAIRMAN: PAST-PRESIDENT H. J. BALL

Committee D-13 on Textile Materials:

Report presented by B. L. Whittier, chairman, and the following actions taken:

Accepted as Tentative:

Specs. and Methods of Test for Industrial Asbestos Tapes (D 1572 - 58 T)

Test for Heat Aging of Asbestos Textiles (D 1573 - 58 T)

Specs. and Methods of Test for Woven Asbestos Cloth (D 1571 - 58 T), to replace Specs. D 677 - 50 and Methods D 577 - 52

Test for Linear Density of Textile Fibers by the Vibroscope (D 1577 - 58 T)

Test for Moisture in Wool by Oven Drying (D 1576 - 58 T)
Test for Extractable Matter in Oven Dried Wool (D 1574 - 58 T)
Test for Fiber Length of Wool (D 1575 - 58 T)
Test for Breaking Strength of Yarn by Skein Method (D 1578 - 58 T)

Accepted as Tentative, Revisions in:

Method and Tolerances for Yarn Containing Wool (D 1285 - 56 T)
Test for Twist in Single Spun Yarns (Untwist-Twist Method) (D 1422 - 56 T)
Rec. Practice for Designation of Yarn Construction (D 1244 - 57 T)
Test for Twist in Yarns (Direct-Counting Method) (D 1423 - 56 T)
Methods of Testing and Tolerances for Tire Cords from Man-Made Fibers (D 885 - 56 T)
Def. of Terms Relating to Textile Materials (D 123 - 55)
Specs. and Methods of Test for Asbestos Tape for Electrical Purposes (D 315 - 52) (Standard reverted to tentative)
Rec. Practice for Designation of Linear Density of Fibers, Yarn Intermediates, Yarns, and Other Textile Materials in the Tex System (D 861 - 52) (Standard reverted to tentative)

Adopted as Standard:

Spec. and Method of Test for Fineness of Wool (D 419 - 55 T)
Test for Fiber Length of Wool Tops (D 519 - 55 T)
Test for Tensile Strength of Wool Fiber Bundles (D 1294 - 56 T)
Test for Wool Content of Raw Wool (Commercial Scale) (D 1334 - 54 T)
Test for Determination of Build-Up and Spread of Glass Yarn as Wrap (Serving) or Braid over Electrical Conductors (D 1332 - 54 T)
Method for Determining the Specific Area and Immaturity Ratio of Cotton Fibers (Arealometer Method) (D 1449 - 55 T)
Rec. Practice for Interlaboratory Testing of Textile Materials (D 990 - 54 T)
Tolerances for Filament Acetate Yarns (D 1235 - 52 T)
Tolerances for Filament Nylon Yarns (D 1236 - 52 T)
Tolerances for Filament Rayon Yarns (D 1237 - 52 T)

Adopted as Standard, Revisions in:

Method of Core Sampling of Raw Wool in Packages for Determination of Percentage of Clean Wool Fiber Present (D 1060 - 56)
Methods of Testing Nonwoven Fabrics (D 1117 - 57)
Def. of Terms Relating to Textile Materials (D 123 - 55)
Specs. and Methods of Test for Asbestos Roving for Electrical Purposes (D 375 - 52)
Specs. and Methods of Test for Asbestos Lap (D 1061 - 57)
Methods of Testing and Tolerances for Glass Yarns (D 578 - 52)
Methods of Testing and Tolerances for Single Jute Yarn (D 541 - 52)
Methods of Testing and Tolerances for Jute Rove and Plied Yarn for Electrical and Packing Purposes (D 681 - 52)
Methods of Testing and Tolerances for Rope Made from Bast and Leaf Fibers (D 738 - 52)
Methods of Testing and Tolerances for Spun, Twisted, or Braided Products Made from Flax, Hemp, Ramie, or Mixtures Thereof (D 739 - 52)
Methods of Testing Single Kraft Yarn (D 1057 - 51)
Methods of Testing Twine Made from Bast and Leaf Fibers (D 1233 - 52)
Test for Fineness of Cotton Fibers by Micronaire (D 1448 - 56)
Test for Water Resistance of Textile Fabrics (D 583 - 54)

Standards Withdrawn:

Method of Testing Woven Asbestos Cloth (D 577 - 52)
Specs. for Woven Asbestos Cloth (D 677 - 50)
Test for Colorfastness to Commercial Laundering and to Domestic Washing of Cotton and Linen Textiles (D 435 - 42)
Test for Colorfastness of Dyed or Printed Wool, Silk, Rayon, or Acetate Fabrics to Laundering or Domestic Washing (D 436 - 37)
Test for Colorfastness to Light of Textiles (D 506 - 55)
Test for Resistance of Textile Fabrics and Yarns to Insect Pests (D 582 - 54)
Test for Evaluating Compounds Designed to Increase Resistance of Fabrics and Yarns to Insect Pests (D 627 - 54)
Test for Colorfastness of Dyed Acetate to Atmospheric Fumes (D 682 - 52)
Test for Resistance of Textile Materials to Microorganisms (D 684 - 57)

Accepted for Publication as Information Only:

Test for Dimensional Change of Knitted Fabrics in Laundering and Dimensional Restorability of the Laundered Fabric.

Papers:

Data Under Difficulties—E. R. Schwarz, presented by the author.
Difficulties with Data—Kenneth Fox and R. D. Wells, presented by Mr. Wells.

FIFTEENTH SESSION—ROAD AND PAVING MATERIALS

TUESDAY, JUNE 24, 2:30 P.M.

SESSION CHAIRMAN: VICE-PRESIDENT K. B. WOODS

Objective Appraisal of Stripping of Asphalt from Aggregate—A. B. Brown, J. W. Sparks, and G. E. Marsh, presented by Mr. Brown.[11]

Field Experience and Laboratory Study of Slow-Setting Paving Grade Asphalt in Airfield Pavement Construction—D. D. Fenton, presented by R. A. Chisholm.

A Laboratory Method for Determining the Skidding Resistance of Bituminous Paving Mixtures—J. W. Shupe and W. H. Goetz, presented by Mr. Shupe.

Effect of Ultraviolet Light on Thin Films of Paving-Grade Asphalts—R. F. Sparlin and Ken R. White, presented by N. W. McLeod.

Suitability of Lightweight Aggregate to the Manufacture of Bituminous Plant Mix—J. C. Wycoff, presented by the author.[12]

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[11] Published in *ASTM STP No. 240.*

[12] Published in the ASTM BULLETIN, No. 235, Jan., 1959, p. 33 (TP 3).

SIXTEENTH SESSION—COMMITTEE REPORT SESSION

TUESDAY, JUNE 24, 4:15 P.M.

SESSION CHAIRMAN: L. L. WYMAN

Committee A-5 on Corrosion of Iron and Steel:

Report presented by Marc Darrin, chairman, and the following actions taken:

Accepted as Tentative:

Test for Weight of Coating on Aluminum-Coated Iron or Steel Articles (A 428 – 58 T)

Accepted as Tentative, Revisions in:

Spec. for Zinc-Coated (Galvanized) Iron or Steel Sheets, Coils and Cut Lengths (A 93 – 55 T)

Spec. for Long Terne Iron or Steel Sheets, Coils and Cut Lengths (A 308 – 53 T)

Spec. for 1.25-oz Class Coating (Pot Yield) Zinc-Coated (Galvanized) Iron or Steel Roofing Sheets (A 361 – 55 T)

Adopted as Standard:

Spec. for Zinc-Coated Steel Wire Strand "Galvanized" and Class A ("Extra Galvanized") (A 122 – 54 T)

Spec. for Zinc-Coated Steel Wire Strand (Class B and Class C Coatings) (A 218 – 54 T)

Spec. for Wire Strand, Overhead Ground, Zinc-Coated Steel (A 363 – 55 T)

Spec. for Poultry Netting (Hexagonal and Straight Line) and Woven Poultry Fencing, Zinc-Coated (Galvanized) Steel (A 390 – 56 T)

Spec. for Tie Wires, Zinc-Coated (Galvanized) Steel (A 112 – 55 T)

Adopted as Standard, Revisions in:

Test for Local Thickness of Electrodeposited Coatings (A 219 – 54) (Jointly with Committee B-8)

Committee A-6 on Magnetic Properties:

Report presented by A. C. Beiler, chairman, and the following actions taken:

Adopted as Standard:

Test for Alternating-Current Magnetic Properties of Laminated Core Specimens (A 346 – 58) (This recommendation was inadvertently indicated in the preprinted report for publication as tentative.)

Adopted as Standard, Revisions in:

Test for Alternating-Current Core Loss, and Permeability of Magnetic Materials (A 343 – 54). This revision was not published since the committee found it necessary to make a further complete revision of the specification, which will be submitted to the Society within the year.

Committee A-7 on Malleable Iron Castings:

Report presented by L. C. Marshall, vice-chairman, and accepted as a report of progress.

The committee withdrew from the report as preprinted the recommendation for the adoption as standard of Tentative Specification for Pearlitic Malleable Iron Castings (A 220 – 55 T).

Committee A-10 on Iron-Chromium, Iron-Chromium-Nickel, and Related Alloys:

Report presented by Jerome Strauss, chairman, and the following actions taken:

Accepted as Tentative:

Specs. for Hot-Rolled and Cold-Finished Corrosion-Resisting Chromium-Nickel-Manganese Steel Bars (A 429 – 58 T)

Accepted as Tentative, Revisions in:

Spec. for Corrosion-Resisting Chromium and Chromium-Nickel Steel Plate, Sheet, and Strip for Fusion-Welded Unfired Pressure Vessels (A 240 – 57 T)

Adopted as Standard, Revisions in:

Spec. for Corrosion-Resisting Chromium-Nickel Steel Plate, Sheet, and Strip (A 167 – 54)
Spec. for High-Strength Corrosion-Resisting Chromium-Nickel Steel Sheet and Strip (A 177 – 54)
Spec. for Corrosion-Resisting Steel Billets and Bars for Reforging (A 314 – 55)

Committee B-6 on Die-Cast Metals and Alloys:

Report presented by W. Babington, chairman, and the following actions taken:

Accepted as Tentative, Revisions in:

Spec. for Aluminum-Base Alloy Die Castings (B 85 – 57 T)

Adopted as Standard, Revisions in:

Spec. for Magnesium-Base Alloy Die Castings (B 94 – 57)

Committee C-4 on Clay Pipe:

Report presented by D. G. Miller, vice-chairman, and accepted as a report of progress.

Committee C-7 on Lime:

Report presented by J. A. Murray, chairman, and the following actions taken:

Accepted as Tentative:

Specs. for Quicklime and Hydrated Lime for Sand-Lime Products (C 415 – 58 T)

Adopted as Standard, Revisions in:

Methods of Physical Testing of Quicklime and Hydrated Lime (C 110 – 57)
Methods of Chemical Analysis of Limestone, Quicklime, and Hydrated Lime (C 25 – 47)

Committee D-22 on Methods of Atmospheric Sampling and Analysis:

Report presented in the absence of the chairman by Morris Katz, vice-chairman, and the following actions taken:

Accepted as Tentative:

Test for Nitrogen Dioxide and Nitric Oxide Content of the Atmosphere (Modified Griess-Ilosvay Reaction) (D 1607 – 58 T), revised to include a procedure for determining nitric oxide.
Test for Oxides of Nitrogen in Gaseous Combustion Products (Phenol-Disulfonic Acid Procedure) (D 1608 – 58 T)
Rec. Practice for Sampling Atmospheres for Analysis of Gases and Vapors (D 1605 – 58 T)
Test for Inorganic Fluoride in the Atmosphere (D 1606 – 58 T)
Test for Oxidant Content of the Atmosphere (D 1609 – 58 T), revised editorially.

Accepted as Tentative, Revisions in:

Method for Continuous Analysis and Automatic Recording of Sulfur Dioxide Content of the Atmosphere (D 1355 – 55 T)
Def. of Terms Relating to Atmospheric Sampling and Analysis (D 1356 – 55 T)

Committee E-6 on Methods of Testing Building Constructions:

Report presented by R. F. Legget, chairman, and accepted as a report of progress.

Committee F-1 on Materials for Electron Tubes and Semiconductor Devices:

Report presented in the absence of the chairman by C. L. Guettel, and the following actions taken:

Accepted as Tentative:

Spec. for Aluminum Oxide Powder (F 7 – 58 T)

SEVENTEENTH SESSION—GILLETT MEMORIAL LECTURE[13]

TUESDAY, JUNE 24, 5:00 P.M.

SESSION CHAIRMAN: PRESIDENT R. T. KROPF

Seventh Gillett Memorial Lecture:

President Kropf opened the session by giving a brief statement of the purpose of the lecture. This lecture is sponsored jointly by the ASTM and the Battelle Memorial Inst. to perpetuate the memory of Horace W. Gillett, one of America's leading technologists, the first Director of the Institute, and for many years a very active worker in the Society, through the presentation of a lecture on a subject pertaining to the development, testing, evaluation, and application of metals.

President Kropf called upon B. D. Thomas, President of Battelle Memorial Inst., who introduced Clyde Williams, President of Clyde Williams and Co., formerly President of Battelle Memorial Inst. Mr. Williams presented the Seventh H. W. Gillett Memorial Lecture on the subject "High-Temperature Metals—Their Role in the Technological Future." The lecturer discussed the needs for materials for high-speed aircraft and missiles. Temperature problems in aircraft result from aerodynamic heating and from the nature of propulsion systems. Each level of speed, each type of engine, each fuel imposes special, and usually rigorous, materials problems. Progress is being made in the development of suitable temperature-resistant materials for airframes and skins, and in the development of truly "high-temperature" alloys and cermets for engine components. Cobalt, columbium, chromium, molybdenum, nickel, and tungsten appear to be the metals most likely to serve as the "workhorses" in the high-temperature age of flight and industrial processing. The demand for some of these metals could readily double or triple by 1965. Evidence exists that there are sufficient supplies of the ores of all these metals readily available to the United States to supply these increased amounts.

President Kropf expressed appreciation to Mr. Williams for his outstanding lecture and presented to him on behalf of the Society the H. W. Gillett Memorial Lecture Certificate.

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[13] To be issued as a separate publication.

EIGHTEENTH SESSION—HIGH TEMPERATURE

TUESDAY, JUNE 24, 8:00 P.M.

SESSION CHAIRMAN: PAUL BRISTER

Effect of Strain-Rate and Temperature on the Strength of Magnesium Alloys—R. W. Fenn, Jr. and J. A. Gusack, presented by Mr. Fenn.

Aging Characteristics of Nickel-Chromium Alloys Containing Appreciable Amounts of Titanium and Aluminum—N. J. Grant and N. E. Rogen, presented by Mr. Rogen.

High-Temperature Properties of Nickel-Al_2O_3 Alloys—W. S. Cremens and N. J. Grant, presented by Mr. Grant.

A Machine for the Evaluation of High-Temperature Alloys Under Combined Static and Dynamic Stresses—P. E. Hawkes and C. H. Ek, presented by Mr. Ek.[14]

Properties of Cast Iron at Elevated Temperatures (Project SP-2 Sponsored by Joint Committee on Effect of Temperature on the Properties of Metals)—J. R. Kattus and Bryan McPherson, presented by Mr. Kattus.[15]

(*Continued in Twenty-Second Session*)

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[14] Published in the ASTM BULLETIN, No. 235, Jan., 1959, p. 46 (TP 16).

[15] Issued as separate publication *ASTM STP No. 248.*

NINETEENTH SESSION—SYMPOSIUM ON EFFECT OF WATER ON BITUMINOUS PAVING MIXTURES[16]

TUESDAY, JUNE 24, 8:00 P.M.

SESSION CHAIRMAN: C. E. PROUDLEY

Field Observations of the Behavior of Bituminous Pavements as Influenced by Moisture—W. K. Parr, presented by the author.

Relationship of Aggregate Characteristics to the Effect of Water on Bituminous Paving Mixtures—J. M. Rice, presented by the author.

Laboratory and Field Tests on Asphalt Paving Mixtures—E. W. Klinger and J. C. Roediger, presented by Mr. Roediger.

Laboratory Study of Anti-Stripping Additives for Bituminous Materials—P. F. Critz, presented by W. J. Halstead.

Methods of Testing for Water Resistance of Bituminous Paving Mixtures—W. H. Goetz, presented by the author.

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[16] To be issued as separate publication *ASTM STP No. 240.*

TWENTIETH SESSION—CRACK PROPAGATION

TUESDAY, JUNE 24, 8:00 P.M.

SESSION CO-CHAIRMEN: R. E. PETERSON
W. T. LANKFORD

The Response of High Strength Steels in the Range of 180,000 to 300,000 psi to Hydrogen Embrittlement from Cadmium Electroplating—E. P. Klier, B. B. Muvdi, and George Sachs, presented by Mr. Klier.

A Fundamental Charpy Impact Transition Temperature—C. E. Hartbower and G. M. Orner, presented by Mr. Hartbower.

Fracture Strengths Relative to Onset and Arrest of Crack Propagation—G. R. Irwin, J. A. Kies, and H. L. Smith, presented by Mr. Irwin.

Size Effects in Slow Notch Bend Tests of a Ni-Mo-V Steel—J. D. Lubahn and S. Yukawa, presented by Mr. Yukawa.

Correlation of Tests Using the Congruency Principle—J. D. Lubahn, presented by the author.

Film: Vulnerability of a Modern Fighter Wing to Fragmentation—Aeronautics Structures Laboratory, Naval Air Materials Center.

TWENTY-FIRST SESSION—SYMPOSIUM ON MATERIALS RESEARCH FRONTIERS[17]

WEDNESDAY, JUNE 25, 9:30 A.M.

SESSION CO-CHAIRMEN: PRESIDENT R. T. KROPF
W. J. SMITH

Tailoring the Properties of Materials—E. P. Stevenson, presented by the author.

Molecular Engineering—Arthur von Hippel, presented by the author.

Instruments in Materials Research—B. H. Billings, presented by the author.

Materials in the Nuclear Age—J. J. Antal, presented by the author.

(*Continued in Twenty-Fourth Session*)

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[17] To be issued as separate publication *ASTM STP No. 243.*

TWENTY-SECOND SESSION—HIGH TEMPERATURE

(Continued from the Eighteenth Session)

WEDNESDAY, JUNE 25, 9:30 A.M.

SESSION CHAIRMAN: H. C. CROSS

Papers:

Temperature and Time Stability of M257 and SAP Aluminum-Aluminum Oxide Alloys—W. S. Cremens, E. A. Bryan, and N. J. Grant, presented by Mr. O. Preston.

The Effect of Atmosphere on Creep-Rupture Properties of a Nickel-Chromium-Aluminum Alloy—Paul Shahinian and M. R. Achter, presented by Mr. Shahinian.

The Creep Properties of Three Low-Shrinkage, Copper-Base Casting Alloys (Sponsored by Joint Committee on Effect of Temperature on the Properties of Metals)—W. F. Simmons and J. G. Kura, presented by Mr. Simmons.

Mechanical and Physical Properties of Three Low-Shrinkage, Copper-Base Casting Alloys (Sponsored by Joint Committee on Effect of Temperature on the Properties of Metals)—J. G. Kura and R. M. Lang, presented by Mr. Kura.

Prediction of Long-Time Creep with Ten-Year Creep Data on Four Plastic Laminates—W. N. Findley and D. B. Peterson, presented by Mr. Findley.

Joint Committee on Effect of Temperature on the Properties of Metals:

Report presented by H. C. Cross, secretary, and the following actions taken:

Accepted as Tentative:

Rec. Practice for Conducting Creep and Time-For-Rupture Tension Tests of Materials (E 139 - 58 T)

Accepted as Tentative, Revisions in:

Rec. Practice for Short-Time Elevated-Temperature Tension Tests of Metallic Materials (E 21 - 43) (Standard reverted to tentative)

Standard Withdrawn:

Rec. Practice for Conducting Long-Time High-Temperature Tension Tests of Metallic Materials (E 22 - 41)

Tentative Withdrawn:

Rec. Practice for Conducting Time-for-Rupture Tension Tests of Metallic Materials (E 85 - 50 T)

SYMPOSIUM ON STABILITY OF DISTILLATE FUEL OILS[18]

WEDNESDAY, JUNE 25

Sponsored by Technical Committee E on Burner Fuel Oils of Committee D-2 on Petroleum Products and Lubricants

SESSION CHAIRMAN: W. DE B. BERTOLETTE

This symposium was arranged in order to review the present situation with regard to the stability of home heating fuel oil and its effect on performance. This review provided background information for a decision by ASTM Technical Committee E as to whether this is a potential category for activity by a subcommittee. The following papers were presented:

Burning Today's Fuel Oils—G. T. Kaufman.

Trials and Tribulations of a Large Development Oil Heating—N. M. Edmands.

Predictive Type Test for Storage Stability and Compatibility of Diesel Fuels—R. T. Jones and J. W. MacDonald.

Requirements for Dependable Performance of Domestic Oil Burner Nozzles—E. O. Olson.

Do We Need a Stability Specification for No. 2 Heating Oil?—Robert Gray.

A Review of the Distillate Fuel Stability Problem.—E. A. Elmquist.

How Distillate Fuel Stability is Measured and Controlled—W. L. Clinkenbeard.

Distillate Fuel Incompatibility—C. C. Ward and F. G. Schwartz.

Distillate Fuel Oil Gel—F. R. Dunn, Jr. and R. W. Sauer.

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[18] To be issued as separate publication *ASTM STP No. 244.*

TWENTY-THIRD SESSION—COMMITTEE REPORT SESSION

WEDNESDAY, JUNE 25, 11:15 A.M.

SESSION CHAIRMAN: G. R. GOHN

Committee B-1 on Wires for Electrical Conductors:

Report presented in the absence of the chairman by C. E. Topping, and the following actions taken:

Accepted as Tentative:

Spec. for Half-Hard Aluminum Wire for Electrical Purposes (B 323 - 58 T)
Spec. for Rectangular and Square Bare Aluminum Wire for Electrical Conductors (B 324 - 58 T)

Accepted as Tentative, Revisions in:

Spec. for Concentric-Lay-Stranded Aluminum Conductors, Steel Reinforced (ACSR) (B 232 - 57 T)

Adopted as Standard:

Spec. for Tinned Soft or Annealed Copper Wire for Electrical Purposes (B 33 - 56 T)
Spec. for Lead-Coated and Lead-Alloy-Coated Soft Copper Wire for Electrical Purposes (B 189 - 56 T)
Spec. for Hard-Drawn Aluminum Wire for Electrical Purposes (B 230 - 55 T)
Spec. for Tinned Hard-Drawn and Medium-Hard-Drawn Copper Wire for Electrical Purposes (B 246 - 56 T)
Method for Determination of Cross-Sectional Area of Stranded Conductors (B 263 - 56 T)

Adopted as Standard, Revisions in:

Spec. for Soft Rectangular and Square Bare Copper Wire for Electrical Conductors (B 48 -57)
Spec. for Rope-Lay-Stranded Copper Conductors Having Bunch-Stranded Members, for Electrical Conductors (B 172 - 55)
Spec. for Rope-Lay-Stranded Copper Conductors Having Concentric-Stranded Members, for Electrical Conductors (B 173 - 55)
Spec. for Bunch-Stranded Copper Conductors for Electrical Conductors (B 174 - 55)
Test for Resistivity of Electrical Conductor Materials (B 193 - 57)
Spec. for Concentric-Lay-Stranded Aluminum Conductors, Hard Drawn and Three-Quarter Hard-Drawn (B 231 - 57)
Spec. for Standard Weight Zinc-Coated (Galvanized) Steel Core Wire for Aluminum Conductors, Steel Reinforced (ACSR) (B 245 - 55)
Spec. for Zinc-Coated (Galvanized) Steel Core Wire (With Coatings Heavier than Standard Weight) for Aluminum Conductors, Steel Reinforced (ACSR) (B 261 - 55)
Classification of Coppers (B 224 - 52) (Jointly with Committees B-2 and B-5)

Committee B-5 on Copper and Copper Alloys, Cast and Wrought:

Report presented by G. H. Harnden, chairman, and the following actions taken:

Accepted as Tentative, Revisions in:

Spec. for General Requirements for Wrought Copper and Copper-Alloy Plate, Sheet, Strip, and Rolled Bar (B 248 - 55 T)
Spec. for General Requirements for Wrought Copper and Copper-Alloy Rod, Bar, and Shapes (B 249 - 55 T)
Spec. for General Requirements for Wrought Copper and Copper-Alloy Wire (B 250 - 55 T)
Spec. for General Requirements for Wrought Seamless Copper and Copper-Alloy Pipe and Tube (B 251 - 55 T)
Spec. for Seamless Tube for Refrigeration Field Service (B 280 - 55 T)
Spec. for Copper-Zinc-Manganese Alloy (Manganese Brass) Sheet and Strip (B 291 - 55 T)
Spec. for Tellurium Copper Rod (B 301 - 55 T)
Spec. for Copper-Silicon Alloy Seamless Pipe and Tube (B 315 - 57 T)

Adopted as Standard, Revisions in:

Spec. for Copper Plates for Locomotive Fireboxes (B 11 - 55)
Spec. for Copper Rods for Locomotive Staybolts (B 12 - 55)
Spec. for Seamless Copper Boiler Tubes (B 13 - 55)
Spec. for Free-Cutting Brass Rod, Bar, and Shapes for Use in Screw Machines (B 16 - 52)
Spec. for Cartridge Brass Sheet, Strip, Plate, Bar, and Disks (B 19 - 55)
Spec. for Naval Brass Rod, Bar, and Shapes (B 21 - 54)
Spec. for Seamless Copper Pipe, Standard Sizes (B 42 - 57)

Spec. for Seamless Red Brass Pipe, Standard Sizes (B 43 – 55)
Spec. for Seamless Copper Tube, Bright Annealed (B 68 – 55)
Spec. for Seamless Copper Tube (B 75 – 57)[19]
Spec. for Seamless Copper Water Tube (B 88 – 55)[19]
Spec. for Copper-Silicon Alloy Plate and Sheet for Pressure Vessels (B 96 – 55)
Spec. for Copper-Silicon Alloy Rod, Bar, and Shapes (B 98 – 55)
Spec. for Rolled Copper-Alloy Bearing and Expansion Plates and Sheets for Bridge and Other Structural Uses (B 100 – 55)
Spec. for Copper and Copper-Alloy Seamless Condenser Tubes and Ferrule Stock (B 111 – 57)
Spec. for Commercial Bronze Strip (B 130 – 55)
Spec. for Seamless Brass Tube (B 135 – 55)[19]
Spec. for Manganese Bronze Rod, Bar, and Shapes (B 138 – 54)
Spec. for Phosphor Bronze Rod, Bar, and Shapes (B 139 – 52)
Spec. for Leaded Red Brass (Hardware Bronze) Rod, Bar, and Shapes (B 140 – 54)
Spec. for Aluminum Bronze Rod, Bar, and Shapes (B 150 – 54)
Spec. for Copper-Nickel-Zinc Alloy (Nickel Silver) Rod and Bar (B 151 – 55)
Spec. for Copper Sheet, Strip, Plate, and Rolled Bar (B 152 – 55)
Test for Expansion (Pin Test) of Copper and Copper-Alloy Tubing (B 153 – 55)
Method of Mercurous Nitrate Test for Copper and Copper Alloys (B 154 – 51)
Spec. for Phosphor Bronze Wire (B 159 – 54)
Spec. for Copper-Alloy Condenser Tube Plates (B 171 – 55)
Spec. for Copper Bus Bar, Rod, and Shapes (B 187 – 55)
Spec. for Seamless Copper Bus Pipe and Tube (B 188 – 56)[19]
Spec. for Silicon Bronze and Silicon Brass Sand Castings (B 198 – 52)
Rec. Practice for Tension Test Specimens for Copper-Base Alloys for Sand Castings (B 208 – 54)
Classification of Coppers (B 224 – 52) (Jointly with Committees B-1 and B-2)
Spec. for Threadless Copper Pipe (B 302 – 57)[19]
Spec. for Copper Drainage Tube (DWV) (B 306 – 57)[19]
Spec. for Copper Rod, Bar, and Shapes (B 133 – 57) (The recommendation for revision of this specification was inadvertently listed in the preprinted report under the heading "Revision of Tentative.")

Committee B-8 on Electrodeposited Metallic Coatings:

Report presented in the absence of the chairman by R. E. Hess, and the following actions taken:

Accepted as Tentative:

Rec. Practice for Cleaning Metals Prior to Electroplating (B 322 – 58 T), with editorial revisions.

Accepted as Tentative, Revisions in:

Spec. for Electrodeposited Coatings of Nickel and Chromium on Steel (A 166 – 55 T)

Adopted as Standard:

Rec. Practice for Preparation of Copper and Copper-Base Alloys for Electroplating (B 281 – 53 T)

Adopted as Standard, Revisions in:

Spec. for Electrodeposited Coatings on Copper and Copper-Base Alloys (B 141 – 55)
Spec. for Electrodeposited Coatings of Nickel and Chromium on Zinc and Zinc-Base Alloys (B 142 – 55)
Test for Local Thickness of Electrodeposited Coatings (A 219 – 54) (Jointly with Committee A-5)

Committee B-9 on Metal Powders and Metal Powder Products:

Report presented in the absence of the chairman by C. G. Johnson, secretary, and the following actions taken:

Accepted as Tentative:

Test for Density and Interconnected Porosity of Sintered Powder Metal Structural Parts and Oil Impregnated Bearings (B 328 – 58 T)
Test for Average Particle Size of Refractory Metals and Compounds by Fisher Subsieve Sizer (B 330 – 58 T)
Test for Apparent Density of Refractory Metals and Compounds by the Scott Volumeter (B 329 – 58 T)
Test for Determination of the Compressibility of Metal Powders (B 331 – 58 T)

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[19] Through an oversight, the revisions of these specifications as printed in the Annual Report of Committee B-5 were incomplete, since no provision was made for the procedure in case of disagreement on the Rockwell hardness values. A sentence has been added to the appropriate section in each specification giving the procedure to be followed.

Accepted as Tentative, Revisions in:

Spec. for Metal Powder Sintered Bearings (Oil Impregnated) (B 202 – 55 T)
Spec. for Sintered Metal Powder Structural Parts from Bronze (B 255 – 54 T)
Spec. for Sintered Metal Powder Structural Parts from Brass (B 282 – 53 T)
Spec. for Copper-Infiltrated Iron Parts (B 303 – 55 T)
Spec. for High Density Iron, Sintered Metal Powder Structural Parts (B 309 – 56 T)
Spec. for Low and Medium Density Iron, Sintered Metal Powder Structural Parts (B 310 – 56 T)
Test for Green Strength of Compacted Metal Powder Specimens (B 312 – 56 T)

Adopted as Standard:

Test for Density of Cemented Carbides (B 311 - 56 T)

Adopted as Standard, Revisions in:

Spec. for Sintered Metal Powder Structural Parts (B 222 – 52)
Def. of Terms Used in Powder Metallurgy (B 243 – 55)

COPPER AND BRASS INDUSTRY LUNCHEON

WEDNESDAY, JUNE 25, 12:00 NOON

TOASTMASTER: GEORGE H. HARNDEN

The luncheon was sponsored by Committees B-2 on Non-Ferrous Metals and Alloys and B-5 on Copper and Copper Alloys, Cast and Wrought.

The guest speaker was R. A. Wilkins, Vice-President, Revere Copper and Brass, Inc., whose subject was "New England Pioneering in Metals and in Methods."

ROAD MATERIALS INDUSTRY LUNCHEON AND AWARDS

WEDNESDAY, JUNE 25, 12:00 NOON

TOASTMASTER: MILES N. CLAIR

The luncheon was sponsored by Committees C-1 on Cement, C-9 on Concrete and Concrete Aggregates, D-4 on Road and Paving Materials, and D-18 on Soils for Engineering Purposes.

The guest speaker was John A. Volpe, President, J. A. Volpe Construction Co., formerly Federal Highway Administrator, whose subject was "Invention, Research, and Science in the Highway Program."[20]

C. A. Hogentogler Award:

The Third C. A. Hogentogler Award was made to Donald M. Burmister, Professor of Civil Engineering, Columbia University, New York City, for his paper on "Applications of Environmental Testing of Soils," published in the 1956 *Proceedings.*

Sanford E. Thompson Award:

The Fourteenth Sanford E. Thompson Award was made to Tien S. Chang, Instructor, and Clyde E. Kesler, Associate Professor, Theoretical and Applied Mechanics, University of Illinois, Urbana, Ill., for their paper on "Correlation of Sonic Properties of Concrete with Creep and Relaxation," published in the 1956 *Proceedings.*

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[20] Published in the ASTM BULLETIN, No. 234, Dec., 1958, p. 7.

TWENTY-FOURTH SESSION—SYMPOSIUM ON MATERIALS RESEARCH FRONTIERS[17]

(*Symposium Continued from the Twenty-first Session*)

WEDNESDAY, JUNE 25, 2:00 P.M.

SESSION CO-CHAIRMEN: VICE-PRESIDENT K. B. WOODS
A. G. H. DIETZ

Recent Advances in Polymer Research—Herman Mark, presented by the author.

Modern Liquid Fuels—A. L. Lyman, presented by the author.

New Advances in Physical Metallurgy—W. R. Hibbard, presented by the author.

Recent Developments in Glass Research—W. W. Shaver, presented by the author.

TWENTY-FIFTH-SESSION—NON-FERROUS METALS

WEDNESDAY, JUNE 25, 2:00 P.M.

SESSION CO-CHAIRMEN: I. V. WILLIAMS
DANIEL CUSHING

Evaluation of a Single Shear Specimen for Sheet Material—W. W. Breindel, C. L. Seale, and R. L. Carlson, presented by Mr. Breindel.

A High Damping Magnesium Alloy for Missile Applications—G. F. Weissmann and W. Babington, presented by Mr. Babington.

A Study of the Variability in the Mechanical Properties of Grade A Phosphor Bronze Strip—M. N. Torrey, G. R. Gohn, and M. B. Wilk, presented by Miss Torrey.

Isochronous Stress-Strain Curves for Some Magnesium Alloys Showing the Effects of Varying Exposure Times on Their Creep Resistance—R. B. Clapper, presented by the author.

Young's Modulus of Magnesium Alloys as a Function of Temperature and Metallurgical Variables—R. W. Fenn, Jr., presented by the author.

TWENTY-SIXTH SESSION—COMMITTEE REPORT SESSION

WEDNESDAY, JUNE 25, 4:00 P.M.

SESSION CHAIRMAN: G. H. HARNDEN

Committee A-1 on Steel:

Report presented in the absence of the chairman by J. W. Caum, and the following actions taken:

Accepted as Tentative:

Specs. for Uncoated Stress-Relieved Wire for Prestressed Concrete (A 421 – 58 T)

Spec. for Wrought Alloy Steel Rolls for Cold and Hot Reduction (A 427 – 58 T)

Specs. for Butt Welds in Still Tubes for Refinery Service (A 422 – 58 T)

Specs. for Seamless and Electric Welded Low Alloy Steel Tubes for Economizers (A 423 – 58 T)

Specs. for Steel Sheets for Porcelain Enameling (A 424 – 58 T)

Specs. for Hot-Rolled Carbon Steel Strip, Commercial Quality (A 425 – 58 T)

Specs. for Centrifugally Cast Ferritic Alloy Steel Pipe for High-Temperature Service (A 426 – 58 T)

Specs. for Factory-Made Wrought Carbon Steel and Alloy Steel Welding Fittings of Seamless or Welded Construction for Low Temperature Service (A 420 – 58 T)

Accepted as Tentative, Revisions in:

Spec. for Steel Bridges and Buildings (A 7 – 57 T)
Spec. for General Requirements for Hot Rolled and Cold Finished Carbon and Alloy Steel Bars (A 29 – 57 T)
Spec. for Welded and Seamless Steel Pipe (A 53 – 58 T)
Spec. for Low-Carbon Steel Track Bolts and Nuts (A 76 – 56 T)
Spec. for Seamless Steel Boiler Tubes (A 83 – 56 T)
Spec. for Forged or Rolled Steel Pipe Flanges, Forged Fittings, and Valves and Parts for High-Temperature Service (A 105 – 57 T)
Spec. for Hot-Rolled Carbon Steel Bars (A 107 – 57 T)
Spec. for Cold-Finished Carbon-Steel Bars and Shafting (A 108 – 57 T)
Spec. for Cold-Rolled Carbon Steel Strip (A 109 – 49 T)
Spec. for Electric-Resistance-Welded Steel Pipe (A 135 – 57 T)
Spec. for Seamless Low-Carbon and Carbon-Molybdenum Steel Still Tubes for Refinery Service (A 161 – 55 T)
Spec. for Electric-Resistance-Welded Steel and Open-Hearth Iron Boiler Tubes (A 178 – 56 T)
Spec. for Seamless Cold-Drawn Low Carbon Steel Heat Exchanger and Condenser Tubes (A 179 – 56 T)
Spec. for Forged or Rolled Steel Pipe Flanges, Forged Fittings, and Valves and Parts for General Service (A 181 – 57 T)
Spec. for Seamless Steel Boiler Tubes for High-Pressure Service (A 192 – 57 T)
Spec. for Alloy-Steel Bolting Materials for High-Temperature Service (A 193 – 57 T)
Spec. for Carbon and Alloy Steel Nuts for Bolts for High-Pressure and High-Temperature Service (A 194 – 57 T)
Spec. for Seamless Cold-Drawn Intermediate Alloy-Steel Heat-Exchanger and Condenser Tubes (A 199 – 56 T)
Spec. for Seamless Intermediate Alloy-Steel Still Tubes for Refinery Service (A 200 – 55 T)
Spec. for Seamless Carbon-Molybdenum Alloy-Steel Boiler and Superheater Tubes (A 209 – 55 T)
Spec. for Medium Carbon Seamless Steel Boiler and Superheater Tubes (A 210 – 55 T)
Spec. for Seamless Alloy Steel Boiler, Superheater, and Heat Exchanger Tubes (A 213 – 57 T)
Spec. for Electric-Resistance-Welded Steel Heat-Exchanger and Condenser Tubes (A 214 – 56 T)
Spec. for Electric-Resistance-Welded Steel Boiler and Superheater Tubes for High-Pressure Service (A 226 – 57 T)
Spec. for Factory-Made Wrought Carbon Steel and Ferritic Alloy Steel Welding Fittings (A 234 – 57 T)
Spec. for Welded Austenitic Stainless Steel Boiler, Superheater, Heat Exchanger, and Condenser Tubes (A 249 – 57 T)
Spec. for Electric-Resistance-Welded Carbon-Molybdenum Alloy-Steel Boiler and Superheater Tubes (A 250 – 55 T)
Spec. for Hot-Rolled Carbon-Steel Strip of Structural Quality (A 303 – 52 T)
Spec. for Steel Machine Bolts and Nuts and Tap Bolts (A 307 – 55 T)
Spec. for Seamless and Welded Austenitic Stainless Steel Pipe (A 312 – 57 T)
Spec. for Alloy-Steel Bolting Materials for Low-Temperature Service (A 320 – 57 T)
Spec. for Quenched and Tempered Steel Bolts and Studs with Suitable Nuts and Plain Hardened Washers (A 325 – 55 T)
Spec. for Seamless and Welded Steel Tubes for Low-Temperature Service (A 334 – 55 T)
Spec. for Ferritic Steel Castings for Pressure Containing Parts Suitable for Low-Temperature Service (A 352 – 57 T)
Spec. for Quenched and Tempered Alloy Steel Bolts and Studs with Suitable Nuts (A 354 – 55 T)
Spec. for Heavy-Walled Carbon and Low Alloy Steel Castings for Steam Turbines (A 356 – 57 T)
Spec. for Structural Steel for Welding (A 373 – 56 T)
Spec. for Factory-Made Wrought Austenitic Steel Welding Fittings (A 403 – 56 T)
Spec. for Welded Large Outside Diameter Light-Wall Austenitic Chromium-Nickel Alloy Steel Pipe for Corrosive or High-Temperature Service (A 409 – 57 T)
Spec. for Carbon Steel Valve Spring Quality Wire (A 230 – 47)
Spec. for Open-Hearth Carbon-Steel Rails (A 1 – 56) (Standard reverted to tentative)
Spec. for Alloy Steel Forgings for Railway Use (A 238 – 55) (Standard reverted to tentative)
Methods of Magnetic Particle Testing and Inspection of Heavy Steel Forgings (A 275 – 47) (In the preprinted Report of Committee A-1, the revisions of these methods were indicated as being for immediate adoption. This was an inadvertence since the methods are being reverted to tentative.)

Adopted as Standard:

Spec. for 5 per cent Chromium, 0.5 per cent Molybdenum Steel Plates for Boilers and Other Pressure Vessels (A 357 – 56 T)
Spec. for Carbon and Alloy Steel Forgings for Pressure Vessel Shells (A 372 – 55 T)
Spec. for Chromium-Silicon Steel Spring Wire (A 401 – 56 T)
Rec. Practice for Selection of Steel Bar Compositions According to Section (A 400 – 56 T)
Spec. for Welded and Seamless Open-Hearth Iron Pipe (A 253 – 55 T)
Spec. for Copper Brazed Steel Tubing (A 254 – 55 T)
Spec. for Metal-Arc Welded Steel Pipe for High-Pressure Transmission Service (A 381 – 54 T)
Spec. for Alloy Steel Chain (A 391 – 56 T)

Adopted as Standard, Revisions in:

Spec. for Mild-Medium-Strength Carbon-Steel Castings for General Application (A 27 – 57)
Spec. for Structural Steel for Locomotives and Cars (A 113 – 56)
Spec. for Structural Steel for Ships (A 131 – 56)
Spec. for Electric-Fusion (Arc)–Welded Steel Pipe (Sizes 4 in. and Over) (A 139 – 55)
Spec. for Structural Rivet Steel (A 141 – 55)
Spec. for High-Strength Steel Castings for Structural Purposes (A 148 – 57)
Spec. for One-Wear Wrought Steel Wheels (A 186 – 54)
Spec. for Seamless and Welded Ferritic Stainless Steel Tubing for General Service (A 268 – 57)
Spec. for Seamless and Welded Austenitic Stainless Steel Tubing for General Service (A 269 – 57)
Spec. for Seamless and Welded Austenitic Stainless Steel Sanitary Tubing (A 270 – 57)
Spec. for Seamless Austenitic Chromium-Nickel Steel Still Tubes for Refinery Service (A 271 – 57)
Spec. for Low and Intermediate Tensile Strength Carbon-Steel Plates of Structural Quality (Plates 2 in. and Under in Thickness) (A 283 – 54)
Spec. for Steel Plates for Pressure Vessels for Service at Low Temperatures (A 300 – 57)
Spec. for Low-Carbon High-Nickel Steel Plate for Pressure Vessels (A 353 – 56)

Withdrawal of Tentatives:

Method of Magnetic Particle Testing and Inspection of Commercial Steel Castings (A 272 – 44 T)
Spec. for High Tensile Strength Carbon-Manganese Steel Plates for Unfired Pressure Vessels (A 397 – 56 T)
Spec. for Chromium-Silicon Steel Spring Wire (A 402 – 56 T)

Committee B-2 on Non-Ferrous Metals and Alloys:

Report presented by Bruce W. Gonser, chairman, and the following actions taken:

Accepted as Tentative:

Spec. for Refined Secondary Lead (B 325 – 58 T)

Accepted as Tentative, Revisions in:

Spec. for Titanium and Titanium Alloy Strip, Sheet, and Plate (B 265 – 52 T)
Spec. for Titanium Sponge (B 299 – 55 T), further revised by the deletion from Table I of grade MD-140.

Adopted as Standard, Revisions in:

Spec. for Slab Zinc (Spelter) (B 6 – 49)
Classification of Coppers (B 224 – 52) (Jointly with Committees B-1 and B-5)

Committee B-3 on Corrosion of Non-Ferrous Metals and Alloys:

Report presented in the absence of the chairman by J. S. Pettibone, and accepted as a report of progress.

Committee D-1 on Paint, Varnish, Lacquer, and Related Products:

Report presented by W. T. Pearce, chairman, and the following actions taken:

Accepted as Tentative:

Specs. for Distilled Linseed Fatty Acids (D 1537 – 58 T)
Specs. for Distilled Soybean Fatty Acids (D 1538 – 58 T)
Specs. for Dehydrated Castor Acids (D 1539 – 58 T)
Test for Effect of Staining Agents on Organic Finishes Used in the Transportation Industry (D 1540 – 58 T)
Test for Total Iodine Value (D 1541 – 58 T)
Test for Color of Transparent Liquids (Gardner Color Scale) (D 1544 – 58 T)
Test for Viscosity by Bubble Time Method (D 1545 – 58 T)

Method for Performance Tests of Clear Floor Sealers (D 1546 – 58 T)
Method for Qualitative Tests for Rosin in Varnishes (D 1542 – 58 T)

Accepted as Tentative, Revisions in:

Def. of Terms Relating to Paint, Varnish, Lacquer, and Related Products (D 16 – 57)
Test for Total Rosin Acids Content of Coating Vehicles (D 1469 – 57 T)
Methods of Testing Varnishes (D 154 – 57 T), comprising method of panel preparation for measurement of gloss of varnishes, and test for print resistance of varnishes.
Specs. for Raw Linseed Oil (D 234 – 55) (Standard reverted to tentative)
Specs. for Boiled Linseed Oil (D 260 – 55) (Standard reverted to tentative)
Specs. for Copper Phthalocyanine Blue (D 963 – 49) (Standard reverted to tentative)

Adopted as Standard:

Specs. for Degummed Soybean Oil (D 124 – 56 T)
Specs. for Dehydrated Castor Oil (D 961 – 55 T)
Specs. for Safflower Oil (D 1392 – 56 T)
Test for Distillation Range of Lacquer Solvents and Diluents (D 1078 – 56 T)
Test for Nonvolatile Content of Resin Solutions (D 1259 – 56 T)
Test for Spectrophotometric Diene Value of Dehydrated Castor Oil and Its Derivatives (D 1358 – 55 T)
Test for Fire Retardance of Paints (Cabinet Method) (D 1360 – 55 T)
Test for Fire Retardance of Paints (Stick and Wick Method) (D 1361 – 55 T)
Test for Alcohol Content of Ketones (D 1362 – 57 T)
Test for Permanganate Time of Lacquer Solvents and Diluents (D 1363 – 57 T)
Test for Water in Lacquer Solvents and Diluents (Fischer Reagent Titration Method) (D 1364 – 55 T)
Test for Abrasion Resistance of Clear Floor Coatings (D 1395 – 56 T)
Methods for Chemical Analysis of Poly(Vinyl Butyral) (D 1396 – 56 T)
Test for Fatty Acid Content of Alkyd Resins and Resin Solutions (D 1398 – 56 T)
Test for Unsaponifiable Content of Tricresyl Phosphate (D 1399 – 56 T)
Methods of Testing Asphalt Emulsions for Use as Protective Coatings for Metal (D 1010 – 55 T) (Jointly with Committee D-8)
Methods for Measurement of Dry Film Thickness of Non-Metallic Coatings of Paint, Varnish, Lacquer, and Related Products Applied on a Non-Magnetic Base (D 1400 – 56 T)
Specs. for Acetone (D 329 – 55 T)
Specs. for Methyl Isobutyl Ketone (D 1153 – 55 T)

Adopted as Standard, Revisions in:

Methods of Testing Drying Oils (D 555 – 54)
Test for Viscosity of Paints, Varnishes, and Lacquers by Ford Viscosity Cup (D 1200 – 54)
Test for Sieve Analysis of Glass Spheres (D 1214 – 54)
Methods of Sampling and Testing Lacquer Solvents and Diluents (D 268 – 53)
Specs. for Ethyl Acetate (85 to 88 per cent Grade) (D 302 – 33)
Specs. for Normal Butyl Acetate (88 to 92 Per Cent Grade) (D 303 – 40)
Specs. for Butanol (Normal Butyl Alcohol) (D 304 – 45)
Specs. for Amyl Acetate (Synthetic) (85 to 88 Per Cent Grade) (D 318 – 39)
Specs. for Amyl Alcohol (Synthetic) (D 319 – 40)
Specs. for Amyl Acetate Made from Fusel Oil (85 to 88 Per Cent Grade) (D 554 – 39)
Specs. for Dibutyl Phthalate (D 608 – 43)
Specs. for Isopropyl Acetate (D 657 – 44)
Specs. for Isopropyl Alcohol (D 770 – 46)
Specs. for Secondary Butyl Acetate (85 to 88 Per Cent Grade) (D 966 – 52)
Specs. for Secondary Butyl Alcohol (D 1007 – 52)
Specs. for Methanol (Methyl Alcohol) (D 1152 – 54)

Standard Withdrawn:

Test for Light Sensitivity of Traffic Paint (D 712 – 47)

Committee D-9 on Electrical Insulating Materials:

Report presented by H. K. Graves, chairman, and the following actions taken:

Accepted as Tentative:

Test for Peroxide Number of Mineral Insulating Oils (D 1563 – 58 T)

Adopted as Standard:

Test for Electrical Resistance of Insulating Materials (D 257 – 57 T)

Test for Insulation Resistivity of Electrical Insulating Oils of Petroleum Origin (D 1169 – 54 T)

Test for Corrosive Sulfur in Electrical Insulating Oils (D 1275 – 58 T)

Methods of Testing Varnished Cotton Fabrics and Varnished Cotton Fabric Tapes Used for Electrical Insulation (D 295 – 55 T)

Methods of Testing Glass-Bonded Mica Used as Electrical Insulation (D 1039 – 50 T)

Methods of Testing Electrical Insulating Oils (D 117 – 54 T)

Adopted as Standard, Revisions in:

Methods of Testing Sheet and Plate Materials Used for Electrical Insulation (D 229 – 49)

Methods of Conditioning Plastics and Electrical Insulating Materials for Testing (D 618 – 54)

Test for Power Factor and Dielectric Constant of Electrical Insulating Oils of Petroleum Origin (D 924 – 49)

Spec. for Communication and Signal Pin-Type Lime-Glass Insulators (D 879 – 49)

Standards Withdrawn:

Methods of Testing Glass Spool Insulators (D 550 – 44)

Accepted for Publication as Information Only:

Test for Evaluation of Thermal Stability of Electrical Insulating Coated Fabrics by Curved Electrodes (Dielectric Prooftest Method)

Test for Evaluation of Thermal Stability of Electrical Insulating Coated Fabrics by Curved Electrodes (Dielectric Breakdown Method)

Test for Oxidation Characteristics of Mineral Transformer Oil

The committee withdrew from the report as preprinted the following recommendations: (1) New Standard Methods of Test for Electrical Power Insulators; (2) Revision and reversion to tentative of Standard Specification for Low and Medium Voltage Pin-Type Lime-Glass Insulators (D 730 – 49); (3) Withdrawal of Standard Methods of Testing Pin-Type Lime-Glass Insulators (D 468 – 46); and (4) Reapproval of Standard Methods of Testing Molding Powders Used in Manufacturing Molded Electrical Insulators (D 392 – 38).

Committee E-4 on Metallography:

The report of Committee E-4 had not been preprinted. By special authorization, Mr. L. L. Wyman, chairman, presented recommendations for the revision of Tentative Methods for Determining the Average Grain Size of Metals (E 112 – 55 T) and Tentative Methods of Preparation of Metallographic Specimens (E 3 – 46 T). These recommendations had received unanimous letter ballot approval of Committee E-4.

Joint Committee on Leather:

Report presented in the absence of the chairman by C. W. Mann, and the following actions taken:

Accepted as Tentative:

Method of Conditioning Leather and Leather Products for Testing (D 1610 – 58 T)

Test for Corrosion Produced by Leather in Contact with Metal (D 1611 – 58 T)

TWENTY-SEVENTH SESSION—COMMITTEE REPORT SESSION

WEDNESDAY, JUNE 25, 4:00 P.M.

SESSION CHAIRMAN: W. C. VOSS

Committee C-3 on Chemical Resistant Mortars:

Report presented in the absence of the chairman by W. C. Hansen, and the following actions taken:

Accepted as Tentative:

Test for Working and Initial Setting Times of Chemically Setting Silicate-Type Chemical-Resistant Mortars (C 414 – 58 T)

Test for Absorption and Apparent Porosity of Chemical-Resistant Mortars (C 413 – 58 T)

Committee C-9 on Concrete and Concrete Aggregates:

Report presented by W. H. Price, chairman, and the following actions taken:

Accepted as Tentative:

Test for Abrasion Resistance of Concrete (C 418 – 58 T)

Accepted as Tentative, Revisions in:

Test for Fundamental Transverse, Longitudinal, and Torsional Frequencies of Concrete Specimens (C 215 – 55 T)

Spec. for Packaged, Dry, Combined Materials for Mortar and Concrete (C 387 – 56 T)
Spec. for Raw or Calcined Natural Pozzolans for Use as Admixtures in Portland Cement Concrete (C 402 – 57 T)
Test for Measuring Mortar-Making Properties of Fine Aggregate (C 87 – 52) (Standard reverted to tentative)
Method of Testing Air-Entraining Admixtures for Concrete (C 233 – 55) (Standard reverted to tentative)
Spec. for Waterproof Paper for Curing Concrete (C 171 – 56) (Standard reverted to tentative)
Spec. for Air-Entraining Admixtures for Concrete (C 260 – 54) (Standard reverted to tentative)

Adopted as Standard:

Test for Air Content of Freshly Mixed Concrete by the Volumetric Method (C 173 – 55 T)
Spec. for Ready-Mixed Concrete (C 94 – 55 T)
Test for Bleeding of Concrete (C 232 – 56 T)

Adopted as Standard, Revisions in:

Def. of Terms Relating to Concrete and Concrete Aggregates (C 125 – 56)
Test for Slump of Portland-Cement Concrete (C 143 – 52)
Spec. for Liquid Membrane-Forming Compounds for Curing Concrete (C 309 – 56)

Committee C-18 on Natural Building Stones:

Report presented in the absence of the chairman by S. F. Etris, and the following actions taken:

Accepted as Tentative:

Spec. for Structural Granite (C 422 – 58 T)

Adopted as Standard:

Spec. for Roofing Slate (C 406 – 57 T)
Test for Weather Resistance of Natural Slate (C 217 – 56 T)

Through an inadvertence, the recommendation for adoption as standard of Tentative Method of Test C 217 – 56 T had been omitted from the preprinted Report of Committee C-18.

Committee D-4 on Road and Paving Materials:

Report presented in the absence of the chairman by A. B. Cornthwaite, vice-chairman, and the following actions taken:

Accepted as Tentative:

Test for Density of Soil in Place by the Sand Cone Method (D 1556 – 58 T) (Jointly with Committee D-18)
Test for Moisture-Density Relations of Soils Using a 10-lb Rammer and an 18-in. Drop (D 1557 – 58 T) (Jointly with Committee D-18)
Test for Moisture-Penetration Resistance Relations of Fine-Grained Soils (D 1558 – 58 T) (Jointly with Committee D-18)
Test for Resistance to Plastic Flow of Bituminous Mixtures by Means of the Marshall Apparatus (D 1559 – 58 T)
Test for Resistance to Deformation and Cohesion of Bituminous Mixtures by Means of Hveem Apparatus (D 1560 – 58 T)
Method of Preparing Bituminous Mixtures by Means of California Kneading Compactor (D 1561 – 58 T)

Accepted as Tentative, Revisions in:

Test for Moisture-Density Relations of Soils, Using 5.5-lb Rammer and a 12-in. Drop (D 698 – 57 T) (Jointly with Committee D-18)
Test for Compressive Strength of Bituminous Mixtures (D 1074 – 55 T)
Methods of Testing Emulsified Asphalts (D 244 – 55)
Spec. for Hot-Mixed, Hot-Laid Asphaltic Concrete for Base and Surface Courses (D 947 – 55)
Spec. for Asphaltic Mixtures for Sheet Asphalt Pavements (D 978 – 54)
Spec. for Bituminous Mixing Plant Requirements (D 995 – 55)
Spec. for Fine Aggregate for Sheet Asphalt and Bituminous Concrete Pavements (D 1073 – 54)
Methods of Sampling Stone, Slag, Gravel, Sand, and Stone Block for Use as Highway Materials (D 75 – 48) (Standard reverted to tentative)

Adopted as Standard:

Test for Water in Petroleum Products and Other Bituminous Materials (D 95 – 56 T) (Jointly with Committee D-2)
Rec. Practice for Quantities of Materials for Bituminous Surface Treatments (D 1369 – 55 T)

Adopted as Standard, Revisions in:

Test for Bitumen Content of Paving Mixtures by Centrifuge (D 1097 – 57)
Spec. for Sodium Chloride (D 632 – 43)

Withdrawal of Tentative:

Spec. for Preformed Expansion Joint Fillers for Concrete (Nonextruding and Resilient Types) (D 544 – 56 T)

The committee withdrew from the report as preprinted the recommendation for the withdrawal of Standard Specifications D 57 – 20, D 58 – 37, D 112 – 30, and D 132 – 39.

Committee E-3 on Chemical Analysis of Metals:

Report presented by Arba Thomas, chairman, and the following actions taken:

Accepted as Tentative:

Method for Determination of Arsenic in Fire-Refined Copper (E 62 – 58 T)
Methods for Chemical Analysis of Magnesium and Magnesium-Base Alloys (E 35 – 58 T)

Adopted as Standard:

Methods for Chemical Analysis of Ferro-Alloys (E 31 – 54 T)
Methods for Chemical Analysis of Nickel-Chromium and Nickel-Chromium-Iron Alloys (E 38 – 56 T)
Methods for Chemical Analysis of Slab Zinc (Spelter) (E 40 – 56 T)
Methods for Chemical Analysis of Zinc-Base Die-Casting Alloys (E 47 – 56 T)
Methods for Chemical Analysis of Sulfur in Special Brasses and Bronzes (E 54 – 50 T)
Methods for Chemical Analysis of Copper-Beryllium Alloys (E 106 – 54 T)
Photometric Methods for Chemical Analysis of Aluminum-Base Alloys (E 34 – 56 T)
Photometric Methods for Chemical Analysis of Lead, Tin, Antimony and Their Alloys (E 87 – 56 T)
Method of Sampling Non-Ferrous Metals and Alloys in Cast Form for Determination of Chemical Composition (E 88 – 50 T)

TWENTY-EIGHTH SESSION—MARBURG LECTURE, MEDAL AWARDS

WEDNESDAY, JUNE 25, 4:30 P.M.

SESSION CHAIRMAN: PRESIDENT R. T. KROPF

Charles B. Dudley Medal Award:

The Twenty-eighth Charles B. Dudley Medal Award was made to O. D. Sherby and T. A. Trozera, Research Engineers, and J. E. Dorn, Professor of Physical Metallurgy, University of California, Berkeley, Calif., for their paper on "Effects of Creep Stress History at High Temperatures on the Creep of Aluminum Alloys," published in the 1956 *Proceedings.*

Richard L. Templin Award:

The Twelfth Richard L. Templin Award was made to H. F. Robertson, D. Gross, and J. Loftus, Fire Protection Section, National Bureau of Standards, Washington, D.C., for their paper on "A Method for Measuring Surface Flammability of Materials Using a Radiant Energy Source," published in the 1956 *Proceedings.*

Sam Tour Award:

The Seventh Sam Tour Award was made to W. G. Renshaw and R. A. Lula, Allegheny Ludlum Steel Corp., Research Laboratory, Brackenridge, Pa., for their paper on "The Corrosion Properties of Chromium-Nickel-Manganese Austenitic Stainless Steels," published in the 1956 *Proceedings.*

Edgar Marburg Lecture:

A brief statement of the purpose of the lecture was given by President Kropf. Elmer Walter Pehrson, Chief, Division of Foreign Activities of the U.S. Bureau of Mines, was then introduced by President Kropf as the thirty-second Edgar Marburg Lecturer. Speaking on the subject, "Man and Raw Materials," Mr. Pehrson discussed the fact that in the light of expanding world population and knowledge that mineral deposits are irreplaceable, recurring shortages understandably cause concern over the adequacy of material resources for the future. These anxieties are aggravated by the threat to military security implicit in potential shortages because raw materials are as indispensable in modern warfare as they are in maintaining peacetime prosperity. He spoke on the major political, economic, and technological factors affecting raw-material availability; the geopolitical implications of raw-material resources and production; and the problems confronting industrial nations in their access to raw-material resources in the backward areas of the world. Consideration was given to national and international raw-material policies and the industrial and military power inherent in the resource and potential raw-material strength of the Sino-Soviet Bloc, as well as a contrast of the resource limitations on industrial growth in the United States and the U.S.S.R.

President Kropf expressed appreciation to Mr. Pehrson for his excellent lecture and presented to him on behalf of the Society the Edgar Marburg Lecture Certificate.

ANNUAL DINNER

WEDNESDAY, JUNE 25, 6:45 P.M.

The New England District Council acted as the official host for this meeting. It reserved Symphony Hall for the ASTM Annual Dinner followed by a concert by the world-famous Boston Pops Orchestra under the direction of Arthur Fiedler. Around 930 people attended this function.

TWENTY-NINTH SESSION—SOILS

THURSDAY, JUNE 26, 9:30 A.M.

SESSION CO-CHAIRMEN: H. B. SEED
E. L. SPENCER

Shear Strength and Elastic Properties of Soil-Cement Mixtures Under Triaxial Loading—G. G. Balmer, presented by the author.

Soil Moisture Suction—Its Importance and Measurement—E. Penner, presented by the author.

The Mechanical Behavior of Chemically Treated Granular Soils—R. L. Schiffman and C. R. Wilson, presented by Mr. Schiffman.

Measurement of Soil Pressure on a Laterally Loaded Pile—Hudson Matlock and E. A. Ripperger, presented by Mr. Ripperger.

Specific Gravity of Montmorillonite Clay Constituents—J. C. Shea, presented by the author.

Compressibility of Sands—J. E. Roberts and J. M. de Souza, presented by Mr. Roberts.

THIRTIETH SESSION—CEMENT

THURSDAY, JUNE 26, 9:30 A.M.

SESSION CHAIRMAN: R. R. LITEHISER

Studies on Calcium Sulpho-Aluminate Admixtures for Expansive Cements—A. Klein and G. E. Troxell, presented by Mr. Klein.

Variations in Portland Cement—Stanton Walker and D. L. Bloem, presented by Mr. Walker.

Aeration Cause of False Set in Portland Cement—W. C. Hansen, presented by the author.

THIRTY-FIRST SESSION—SYMPOSIUM ON PARTICLE SIZE MEASUREMENT[22]

THURSDAY, JUNE 26, 9:30 A.M.

SESSION CHAIRMAN: A. E. REED

I. Sieves, Sieving Methods and Microscopy

Introduction—L. T. Work

The Mechanics of Fine Sieving—K. T. Whitby, presented by the author.

A Review of Sieve Standardization—L. V. Judson (presented by title only).

Application of Electroformed Precision Micromesh Sieves to the Determination of Particle Size Distribution of Cracking Catalysts—H. W. Daeschner, E. E. Seibert, and E. D. Peters, presented by Mr. Daeschner.

A Review of the Work of the API Committee on Analytical Research Subcommittee on Measurement of Physical Properties of Cracking Catalysts—L. L. Mittelman, presented by B. J. Heinrich.

The Stanford Research Institute Particle Bank—R. D. Cadle and W. C. Thuman, presented by R. D. Cadle.

(*Continued in Thirty-Third and Thirty-Ninth Sessions*)

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[22] To be issued as separate publication *ASTM STP No. 234*.

THIRTY-SECOND SESSION—COMMITTEE REPORT SESSION

THURSDAY, JUNE 26, 11:15 A.M.

SESSION CHAIRMAN: J. H. KOENIG

Committee C-8 on Refractories:

Report presented in the absence of the chairman by S. F. Etris, and the following actions taken:

Accepted as Tentative:

Test for Thermal Conductivity of Castable Refractories (C 417 – 58 T)
Classification of Silica Refractory Brick (C 416 – 58 T)
Classification of Fireclay and High Alumina Refractory Brick (C 27 – 58 T)

Accepted as Tentative, Revisions in:

Def. of Terms Relating to Refractories (C 71 – 55)
Spec. for Fireclay-Base Castable Refractories for Boiler Furnaces and Incinerators (C 213 – 55)

Adopted as Standard:

Test for Bulk Density of Granular Refractory Materials (C 357 – 55 T)

Adopted as Standard, Revisions in:

Classification of Fireclay Refractories (C 27 – 56)
Method for Basic Procedure in Panel Spalling Test for Refractory Brick (C 38 – 52)
Spec. for Fireclay-Base Castable Refractories for Boiler Furnaces and Incinerators (C 213 – 55)

Tentative Revision Withdrawn:

Def. of Terms Relating to Refractories (C 71 – 55)

Committee C-12 on Mortars for Unit Masonry:

Report presented in the absence of the chairman by S. F. Etris, and accepted as a report of progress.

Committee C-19 on Structural Sandwich Constructions:

Report presented in the absence of the chairman by J. F. Anderson, and accepted as a report of progress.

Committee C-20 on Acoustical Materials:

Report presented in the absence of the chairman by S. F. Etris, and the following actions taken:

Accepted as Tentative:

Test for Sound Absorption of Acoustical Materials in Reverberation Rooms (C 423 – 58 T)

Adopted as Standard:

Test for Impedance and Absorption of Acoustical Materials by the Tube Method (C 384 – 56 T)

Committee C-21 on Ceramic Whitewares and Related Products:

Report presented in the absence of the chairman by S. F. Etris, and the following actions taken:

Accepted as Tentative:

Test for Crazing Resistance of Fired Glazed Whitewares by Autoclave Treatment (C 424 – 58 T)

Accepted as Tentative, Revisions in:

Def. of Terms Relating to Ceramic Whitewares and Related Products (C 242 – 56 T)

Adopted as Standard:

Test for Compressive (Crushing) Strength of Fired Whiteware Materials (C 407 – 57 T)
Test for Thermal Conductivity of Whiteware Ceramics (C 408 – 57 T)

Committee C-22 on Porcelain Enamel:

Report presented in the absence of the chairman by S. F. Etris, and the following actions taken:

Accepted as Tentative, Revisions in:

Def. of Terms Relating to Porcelain Enamel (C 286 – 57 T)

Adopted as Standard:

Test for Thermal Shock Resistance of Porcelain-Enameled Utensils (C 385 – 56 T)
Classification of Water Used in Milling of Porcelain Enamel (C 375 – 55 T)

Committee D-5 on Coal and Coke:

Report presented in the absence of the chairman by R. L. Coryell, vice-chairman, and the following actions taken:

Accepted as Tentative, Revisions in:

Test for Cubic Foot Weight of Crushed Bituminous Coal (D 291 – 29) (Standard reverted to tentative)

Adopted as Standard, Revisions in:

Methods of Laboratory Sampling and Analysis of Coal and Coke (D 271 – 48)

Committee D-14 on Adhesives:

Report presented in the absence of the chairman by S. F. Etris, and the following actions taken:

Accepted as Tentative:

Test for Bonding Permanency of Water or Solvent Soluble Liquid Adhesives for Labeling Glass Bottles (D 1581 – 58 T)
Test for Nonvolatile Content of Phenol, Resorcinol, and Melamine Adhesives (D 1582 – 58 T)
Test for Measuring the Hydrogen Ion Concentration of Dry Adhesive Films (D 1583 – 58 T)
Test for Water Absorptiveness of Paper Labels (D 1584 – 58 T)
Test for Filler Content of Phenol, Resorcinol, and Melamine Adhesives (D 1579 – 58 T)
Spec. for Adhesives for Automatic Machine Labeling of Glass Bottles (D 1580 – 58 T)

Accepted as Tentative, Revisions in:

Test for Effect of Moisture and Temperature on Adhesive Bonds (D 1151 – 56 T)
Def. of Terms Relating to Adhesives (D 907 – 55)

Adopted as Standard, Revisions in:

Test for Resistance of Adhesive Bonds to Chemical Reagents (D 896 – 51)

PETROLEUM INDUSTRY LUNCHEON

THURSDAY, JUNE 26, 12:00 NOON

TOASTMASTER: W. T. GUNN

The luncheon was sponsored by Committee D-2 on Petroleum Products and Lubricants.

The guest speaker was Hoyt C. Hottel, Professor of Fuel Engineering, and Director, Fuels Research Laboratory, Massachusetts Institute of Technology, whose subject was "Oils' Competitors in Space Heating."

INSTRUMENT AND APPARATUS INDUSTRY LUNCHEON

THURSDAY, JUNE 26, 12:00 NOON

CO-TOASTMASTERS: A. C. WEBBER AND D. C. SCOTT, JR.

The luncheon was sponsored by Committee E-1 on Methods of Testing.

The guest speaker was Dr. A. V. Astin, Director, National Bureau of Standards, whose subject was "Instrumentation, Where We Stand."[23]

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[23] Published in the ASTM BULLETIN, No. 232, Sept., 1958, p. 7.

THIRTY-THIRD SESSION—SYMPOSIUM ON PARTICLE SIZE MEASUREMENT[22]

(*Symposium continued from the Thirty-first Session*)

THURSDAY, JUNE 26, 2:00 P.M.

SESSION CHAIRMAN: A. E. JACOBSEN

II. Sedimentation Methods

Recent Developments in the Hydrometer Method as Applied to Soils—E. E. Bauer, presented by the author.

Sedimentation Procedures for Determining Particle Size Distribution—W. F. Sullivan and A. E. Jacobsen, presented by Mr. Sullivan.

A Photoelectric Sedimentation Method for Particle Size Determination in the Sub-Sieve Range—H. R. Harner and J. R. Musgrave, presented by Mr. Musgrave.

Centrifuge Sedimentation Size Analysis of Membrane Filter Collected Samples of Airborne Dusts—K. T. Whitby, A. B. Algren, and J. C. Annis, presented by Mr. Whitby.

A Liquid Sedimentation Method for Particle Size Distributions—L. M. Cartwright and R. Q. Gregg, presented by B. J. Heinrich.

New Methods for Determining Particle Size Distribution by Examining Gravitational and Centrifugal Sedimentation—Soren Berg, presented by V. D. Frechette.

(*Continued in Thirty-Ninth Session*)

THIRTY-FOURTH SESSION—SYMPOSIUM ON PAPER AND PAPER PRODUCTS[24]

THURSDAY, JUNE 26, 2:30 P.M.

SESSION CO-CHAIRMEN: H. A. BIRDSALL
F. F. NEWKIRK

Some Historical Developments in Paper Testing—W. R. Willets and F. R. Marchetti, presented by Mr. Willets.

New Developments in the Internal Bonding of Paper—K. W. Britt and J. W. Eastes, presented by Mr. Britt.

Non-Woven Fabrics and Synthetic Fiber Papers: Technology and End Uses—J. T. Taylor and P. J. McLaughlin, presented by Mr. Taylor.

Relative Humidity Measurements in Packaging Material Testing—B. H. Schrier and P. K. Wolper (presented by title only).

(*Continued in Thirty-Sixth Session*)

THIRTY-FIFTH SESSION—COMMITTEE REPORT SESSION

THURSDAY, JUNE 26, 4:30 P.M.

SESSION CHAIRMAN: W. A. KIRKLIN

Committee B-4 on Metallic Materials for Electrical Heating, Electrical Resistance, and Electrical Contacts:

Report presented by E. I. Shobert, II, chairman, and the following action taken:

Accepted as Tentative:

Test for Resistance Characteristics of Microcontacts (B 326 – 58 T)

Committee B-7 on Light Metals and Alloys, Cast and Wrought:

Report presented by I. V. Williams, chairman, and the following actions taken:

Accepted as Tentative:

Spec. for Aluminum Alloy Hardeners Used in Making Zinc-Base Die Casting Alloys (B 327 – 58 T)

Accepted as Tentative, Revisions in:

Spec. for Aluminum-Base Alloy Sand Castings (B 26 – 57 T)[25]

Spec. for Aluminum-Base Alloy Permanent Mold Castings (B 108 – 57 T)[25]

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[24] To be issued as a separate publication *ASTM STP No. 241*.

[25] A negative vote was cast at the session on the acceptance of the revisions in Specifications B 26 and B 108 with respect to the proposed withdrawal of alloy SC64C.

Spec. for Aluminum Alloy Sheet and Plate (B 209 - 57 T)
Spec. for Aluminum Alloy Drawn Seamless Tubes (B 210 - 57 T)
Spec. for Aluminum-Alloy Bars, Rods, and Wire (B 211 - 57 T), further revised by the addition of a new Section 13 on Cladding Thickness.
Spec. for Aluminum Alloy Extruded Bars, Rods, and Shapes (B 221 - 57 T)
Spec. for Aluminum Alloy Drawn Seamless Tubes for Condensers and Heat Exchangers (B 234 - 57 T)
Spec. for Aluminum Alloy Extruded Tubes (B 235 - 57 T)
Spec. for Aluminum Bars for Electrical Purposes (B 236 - 57 T)
Spec. for Aluminum Alloy Pipe (B 241 - 57 T)
Spec. for Aluminum Alloy Die Forgings (B 247 - 57 T)
Spec. for Aluminum Alloy Drawn Seamless Coiled Tubes for Special Purpose Applications (B 307 - 57 T)
Spec. for Aluminum Alloy Standard Structural Shapes Rolled or Extruded (B 308 - 57 T)
Spec. for Alluminum Alloy Round Welded Tubes (B 313 - 57 T)
Spec. for Aluminum Alloy Rivet and Cold Heading Wire and Rods (B 316 - 57 T)
Spec. for Extruded Aluminum Alloy Bars, Rods, Pipe, and Structural Shapes for Electrical Purposes (B 317 - 57 T)
Spec. for Type A and Type B Aluminum Alloy Drawn Annealed Seamless Coiled Tubes (B 318 - 57 T)

Adopted as Standard:

Spec. for Magnesium-Base Alloy Sheet and Plate (B 90 - 57 T)
Spec. for Magnesium-Base Alloy Sand Castings (B 80 - 57 T)
Spec. for Magnesium-Base Alloy Forgings (B 91 - 57 T)
Spec. for Magnesium-Base Alloys in Ingot Form for Sand Castings, Die Castings, and Permanent Mold Castings (B 93 - 57 T)
Spec. for Magnesium-Base Alloy Bars, Rods, and Shapes (B 107 - 57 T)
Spec. for Magnesium-Base Alloy Permanent Mold Castings (B 199 - 57 T)
Spec. for Magnesium-Base Alloy Extruded Tubes (B 217 - 57 T)

Adopted as Standard, Revisions in:

Spec. for Aluminum-Base Alloys in Ingot Form for Sand Castings, Die Castings, and Permanent Mold Castings (B 179 - 57)
Rec. Practice for Codification of Light Metals and Alloys, Cast and Wrought (B 275 - 55)

Committee D-17 on Naval Stores:

Report presented by V. E. Grotlisch, chairman, and the following actions taken:

Accepted as Tentative:

Test for Fatty Acids Content of Tall Oil Rosin (D 1585 - 58 T), revised by the deletion of Sections 17 through 22 covering Alternate Method B.

Adopted as Standard:

Methods of Testing Tall Oil (D 803 - 55 T)

This recommendation, while not included in the preprinted report of Committee D-17, was presented at the session and accepted since it had received unanimous letter ballot approval in the committee.

Adopted as Standard, Revisions in:

Methods of Sampling and Testing Turpentine (D 233 - 57)
Test for Volatile Oil in Rosin (D 889 - 50)
Test for Water in Liquid Naval Stores (D 890 - 51)
Test for Iron in Rosin (D 1064 - 51)

Committee D-21 on Wax Polishes and Related Materials:

Report presented in the absence of the chairman by M. D. Huber, and accepted as a report of progress.

Committee D-23 on Cellulose and Cellulose Derivatives:

Report presented in the absence of the chairman by M. D. Huber, and the following actions taken:

Accepted as Tentative, Revisions in:

Methods of Testing Sodium Carboxymethylcellulose (D 1439 - 56 T)
Test for Moisture in Cellulose (D 1348 - 56) (Standard reverted to tentative)

Committee D-24 on Carbon Black:

Report presented by S. R. Doner, chairman, and accepted as a report of progress.

Committee E-1 on Methods of Testing:

Report presented, in the absence of the chairman, by P. J. Smith, secretary, and the following actions taken:

Accepted as Tentative:

Specs. for Pensky-Martens Closed Flash Tester (E 134 - 58 T)

Method for Determination of Poisson's Ratio at Room Temperature (E 132 – 58 T)

Accepted as Tentative, Revisions in:

Defs. of Terms Relating to Methods of Mechanical Testing (E 6 – 54 T), with editorial changes in the definition of "Fatigue Strength Reduction Factor" to read "Fatigue Notch Factor," and the Note deleted from the definition.

Specs. for Cell-Type Oven with Controlled Rates of Ventilation (E 95 – 52 T)

Method of Determination of Young's Modulus at Room Temperature (E 111 – 55 T)

Adopted as Standard:

Specs. for Distillation Equipment (E 133 – 58)

Hardness Conversion Tables for Metals (Relationship Between Brinell Hardness, Diamond Pyramid Hardness, Rockwell Hardness, and Rockwell Superficial Hardness) (E 140 – 58) to replace Standards E 33 – 42, E 48 – 47, and E 93 – 52.

Adopted as Standard, Revisions in:

Method of Testing and Standardization of Etched-Stem Liquid-in-Glass Thermometers (E 77 – 56 T)

Spec. for ASTM Thermometers (E 1 – 57), covering (1) immediate adoption of revision, with further revisions to include changes in the requirements for dimensions of thermometers, similar to those given in the report for the precision thermometers, subject to approval of the interested technical committees and letter ballot approval of Committee E-1; and (2) adoption of tentative revisions as standards.

Standards Withdrawn:

Hardness Conversion Table for Cartridge Brass (Relationship Between Diamond Pyramid Hardness, Rockwell Hardness, and Brinell Hardness) (E 33 – 42)

Hardness Conversion Tables for Steel (Relationship Between Diamond Pyramid Hardness, Rockwell Hardness, and Brinell Hardness) (E 48 – 47)

Hardness Conversion Table for Nickel and High Nickel Alloys (Relationship Between Diamond Pyramid Hardness, Brinell Hardness, and Rockwell Hardness) (E 93 – 52)

Committee E-2 on Emission Spectroscopy:

Report presented by D. L. Fry, chairman, and the following actions taken:

Accepted as Tentative:

Def. of Terms Relating to Emission Spectroscopy (E 135 – 58 T)

Accepted for Publication as Information Only:

Method for Spectrochemical Analysis of Caustic Soda by the Solution - A-C Arc Technique.

Committee E-7 on Nondestructive Testing:

Report presented in the absence of the chairman by J. W. Caum, and the following action taken:

Accepted as Tentative:

Method for Wet Magnetic Particle Inspection (E 138 – 58 T)

The committee withdrew from the report as preprinted the recommendation for publication as tentative of Method for Quality Inspection by Radiographic Testing.

Committee E-12 on Appearance:

Report presented from manuscript by George Ingle, chairman, and accepted as a report of progress.

Committee E-13 on Absorption Spectroscopy:

Report presented in the absence of the chairman by R. F. Robey, secretary, and the following action taken:

Accepted for Publication as Information Only:

Methods for Evaluation of Spectrophotometers

THIRTY-SIXTH SESSION—SYMPOSIUM ON PAPER AND PAPER PRODUCTS[24]

(*Symposium Continued from Thirty-fourth Session*)

THURSDAY, JUNE 26, 7:30 P.M.

SESSION CO-CHAIRMEN: H. A. BIRDSALL
F. F. NEWKIRK

Committee D-6 on Paper and Paper Products:

Report presented by R. H. Carter, chairman, and the following actions taken:

Accepted as Tentative, Revisions in:

Test for Thickness of Paper and Paper Products (D 645 - 43) (Standard reverted to tentative)

Adopted as Standard:

Test for Resistance of Paper to Passage of Air (D 726 - 55 T)

Test for Water Resistance of Paper, Paperboard, and Other Sheet Materials by the Dry-Indicator Method (D 779 - 55 T)

Test for Pinholes in Glassine and Other Greaseproof Papers (D 1221 - 52 T)

Adopted as Standard, Revisions in:

Test for Titanium Dioxide in Paper (D 921 - 49)

Tentative Withdrawn:

Test for Water Vapor Permeability of Paper and Paperboard (D 988 - 51 T)

Symposium Papers:

Testing of Synthetic Fiber Papers—F. H. Koontz and J. K. Owens, presented by Mr. Koontz.

A New Cotton Paper Furnish—Properties, Applications, Identification—J. A. Harpham, presented by the author.

Clupak—A Revolutionary High Stretch Paper—Its Manufacture and Performance—R. J. Diaz, presented by the author.

Knit Paper—Its Uses as a Utilitarian Textile—R. H. Marks, presented by C. E. Brandon.

THIRTY-SEVENTH SESSION—SYMPOSIUM ON APPLICATION OF SOIL TESTING IN HIGHWAY DESIGN AND CONSTRUCTION[26]

THURSDAY, JUNE 26, 8:00 P.M.

SESSION CHAIRMAN: C. A. CARPENTER

Examples of Highway Soil Engineering—E. S. Barber, presented by the author.

Subsurface Explorations with Drilling Machines, Power Augers, and Earth Resistivity—D. G. Shurig and E. J. Yoder, presented by Mr. Shurig.

The Soil Exploration and Mapping Cooperative Project in Illinois—Nicholas Chryssafopoulos, presented by the author.

Investigation of Banded Sediments Along St. Lawrence–North Shore in Quebec—R. W. J. Pryer and K. B. Woods, presented by Mr. Pryer.

(*Continued in Forty-Second Session*)

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[26] To be issued as a separate publication *ASTM STP No. 239.*

THIRTY-EIGHTH SESSION—SYMPOSIUM ON RADIOACTIVITY IN INDUSTRIAL WATER AND INDUSTRIAL WASTE WATER[27]

THURSDAY, JUNE 26, 8:00 P.M.

SESSION CHAIRMAN: A. R. BELYEA

Presentation of Max Hecht Award:

The Fifth Max Hecht Award was given to F. R. Owens, Cyrus William Rice and Co., Inc., Pittsburgh, Pa., who has performed outstanding work in Committee D-19 and in the field of industrial water.

Committee D-19 on Industrial Water:

Report presented by Max Hecht, chairman, and the following actions taken:

Accepted as Tentative:

Test for Dissolved and Gaseous Hydrogen in Industrial Water (D 1588 - 58 T), revised by the deletion of Figs. 2 and 3.
Test for Dissolved Oxygen in Industrial Waste Water (D 1589 - 58 T)
Test for Effect of Water in Tubular Heat Exchangers (D 1591 - 58 T)
Test for Surface Tension of Industrial Water and Industrial Waste Water (D 1590 - 58 T)

Accepted as Tentative, Revisions in:

Test for Phosphate in Industrial Water (D 515 - 55 T)
Method for Identification of Types of Microorganisms in Industrial Water (D 1128 - 50 T)
Test for Chemical Oxygen Demand (Dichromate Oxygen Demand) of Industrial Waste Water (D 1252 - 55 T)
Test for Odor of Industrial Waste Water (D 1292 - 53 T)
Test for Hydrazine in Industrial Water (D 1385 - 57 T)

Adopted as Standard:

Test for Suspended and Dissolved Matter (Suspended and Dissolved Solids) in Industrial Water and Industrial Waste Water (D 1069 - 54 T)
Test for pH of Industrial Water and Industrial Waste Water (D 1293 - 55 T)
Test for Ammonia in Industrial Water and Industrial Waste Water (D 1426 - 56 T)
Test for Residual Chlorine in Industrial Waste Water (D 1427 - 56 T)

The committee withdrew from the report as preprinted the recommendation for adoption as standard of Tentative Method of Test for Evaluating Acute Toxicity of Industrial Waste Water to Fresh Water Fishes (D 1345 - 54 T)

Adopted as Standard, Revisions in:

Method of Reporting Results of Analysis of Industrial Water and Industrial Waste Water (D 596 - 55)
Test for Iron Bacteria in Industrial Water and Water-Formed Deposits (D 932 - 51)
Test for Sulfate-Reducing Bacteria in Industrial Water and Water-Formed Deposits (D 993 - 51)
Def. of Terms Relating to Industrial Water and Industrial Waste Water (D 1129 - 57)
Test for Fluoride Ion in Industrial Water and Industrial Waste Water (D 1179 - 57)

Symposium Papers:

Introduction—A. R. Belyea.
Radioactivity and Purity Control of APPR Primary Water—A. L. Medin, presented by the author.
Radioactive Waste Processing and Control at the Shippingport Atomic Power Station—S. F. Whirl and J. A. Tash, presented by Mr. Tash.
Test Methods for Radiation Hazards in Industrial Water—C. J. Munter, presented by the author.
Analysis for Radionuclides in Aqueous Wastes from an "Atomic" Plant—B. Kahn, D. W. Moeller, T. H. Handley, and S. A. Reynolds, presented by Mr. Reynolds.
The Analysis of Environmental Samples for Radionuclides—D. L. Reid, presented by the author.
Open Discussion.
Summation—J. M. Seamon.

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[27] To be issued as a separate publication *ASTM STP No. 235.*

THIRTY-NINTH SESSION—SYMPOSIUM ON PARTICLE SIZE MEASUREMENT[22]

(Symposium Continued from Thirty-first and Thirty-third Sessions)

FRIDAY, JUNE 27, 9:30 A.M.

SESSION CHAIRMAN: L. T. WORK

III. Other Particle Size Methods and Correlations

A Discussion of the Recommended Practice of Reporting Particle Size Characteristics of Pigments (ASTM Tentative Method D 1366 – 55 T)—J. H. Calbeck, presented by the author.

The Determination of Particle Size by Adsorption Methods—R. J. Fries, presented by the author.

A Study of the Blaine Fineness Tester. Calculation of Surface Area from Air Permeability Data—S. S. Ober and K. J. Frederick, presented by Mr. Ober.

Three-Dimensional Electronic Sizing of Sub-Sieve Particles at 6000 per Second—R. H. Berg, presented by the author.

Turbidimetric Particle Size Distribution Theory: Application to Refractory Metal and Oxide Powders—A. I. Michaels, presented by the author.

FORTIETH SESSION—COMMITTEE REPORT SESSION

FRIDAY, JUNE 27, 12:30 P.M.

SESSION CHAIRMAN: A. E. JUVE

Committee A-3 on Cast Iron:

Report presented by D. E. Krause, chairman, and the following actions taken:

Adopted as Standard:

Methods of Chill Testing of Cast Iron (A 367 – 55 T)
Spec. for High-Strength Nodular Iron Castings (A 396 – 55 T)

Adopted as Standard, Revisions in:

Spec. for Automotive Gray Iron Castings (A 159 – 55)

Committee D-2 on Petroleum Products and Lubricants:

Report presented by H. M. Smith, chairman, and the following actions taken:

Accepted as Tentative:

ASTM Butadiene Measurement Tables (D 1550 – 58 T)
Test for Zinc in New Lubricating Oils and Lubricating Oil Additives (Polarographic Method) (D 1549 – 58 T)
Test for Vanadium in Navy Special Fuel Oil (D 1548 – 58 T)
Test for Sulfur in Petroleum Oils by Quartz Tube Combustion (D 1551 – 58 T)
Methods for Analysis of Graphites Used as Lubricants (D 1553 – 58 T)
Test for Sulfur in Petroleum Products by the Rapid, High-Temperature Combustion Method (D 1552 – 58 T)
Method for Calculation of Volume and Weight of Benzene, Toluene, and Paraxylene (D 1555 – 58 T) (Jointly with Committee D-16)
Method of Test for Autogenous Ignition Temperatures of Petroleum Products (D 286 – 30) (Standard reverted to tentative)

Accepted as Tentative, Revisions in:

Test for Water and Sediment by Means of Centrifuge (D 96 – 57 T)
Test for Dilution of Crankcase Oils (D 322 – 55 T)
Test for Existent Gum in Fuels by Jet Evaporation (D 381 – 57 T)
Specs. for Gasoline (D 439 – 57 T)
Test for Carbon Residue of Petroleum Products, Ramsbottom Coking Method (D 524 – 52 T)
Test for Ignition Quality of Diesel Fuels by the Cetane Method (D 613 – 56 T)
Test for Knock Characteristics of Aviation Fuels by the Aviation Method (D 614 – 56 T)
Test for Foaming Characteristics of Lubricating Oils (D 892 – 46 T)
Test for Knock Characteristics of Aviation Fuels by the Supercharge Method (D 909 – 49 T)
Test for Neutralization Value by Color-Indicator Titration (D 974 – 55 T), with an editorial change in the Specs. for *p*-naphtholbenzein, changing "benzene" to toluene in Section 3 (2 places) and Section 4(*c*)

Test for Olefinic Plus Aromatic Hydrocarbons in Petroleum Distillates (D 1019 – 57 T)
Test for Phosphorus in Lubricating Oils, Additives, and Concentrates (D 1091 – 54 T)
Method for Calibrating Upright Tanks (D 1220 – 57 T)
Test for Hydrocarbon Types in Liquid Petroleum Products (Fluorescent Indicator Adsorption (FIA) Method) (D 1319 – 56 T)
Test for Smoke Point of Jet Fuels (D 1322 – 54 T)
Test for Trace Concentrations of Tetraethyllead in Primary Reference Fuels (D 1368 – 55 T)
Method for Calibrating Spherical and Spheroidal Tanks (D 1408 – 56 T)
Test for Flash Point by Pensky-Martens Closed Tester (D 93 – 52) (Standard reverted to tentative)
Test for Apparent Viscosity of Lubricating Greases (D 1092 – 55) (Standard reverted to tentative)

Adopted as Standard:

Test for Water in Petroleum Products and Other Bituminous Materials (D 95 – 56 T) (Jointly with Committee D-4)
Test for Penetration of Petrolatum (D 937 – 49 T)
Method for Polarographic Determination of Tetraethyllead in Gasoline (D 1269 – 56 T)
Test for Effect of Copper on Oxidation Rate of Grease (D 1402 – 56 T)

Adopted as Standard, Revisions in:

Test for Sulfur in Petroleum Products and Lubricants by the Bomb Method (D 129 – 57)
Test for Carbon Residue of Petroleum Products (Conradson Carbon Residue) (D 189 – 52)
Test for Vapor Pressure of Petroleum Products (Reid Method) (D 323 – 56)
Test for Knock Characteristics of Motor Fuels by the Motor Method (D 357 – 56)
Test for Neutralization Value (Acid and Base Numbers) of Petroleum Products (Potentiometric Titration Method) (D 664 – 54)
Test for Knock Characteristics of Motor Fuels by the Research Method (D 908 – 56)

Accepted for Publication as Information Only:

Test for Dilution of Automotive Crankcase Oils (Reduced Pressure Distillation Method)
Method of Computing ASTM Butadiene Measurement Tables
Test for Filterability of Aviation Turbine Fuels
Test for Determination of Particulate Matter in Hydrocarbons
Test for Sealing Strength of Paraffin Wax
Test for Breaking Strength of Petroleum Waxes
Test for Hydrocarbon Types in Low Olefinic Gasoline by Mass Spectrometry
Test for Maximum Fluidity Temperature of Residual Fuel Oil
Test for Ash Content of Petroleum Oils
Test for Knock Characteristics of Motor Fuels Above 100 Octane Number by the Research Method
Specs. for Aviation Turbine Fuels
Test for Thermal Stability of Aviation Turbine Fuels
Optional Method for Analysis of Lubricating Greases (to be published as an appendix to Method D 128)

The committee withdrew from the report as preprinted the following recommendations: (1) Proposed new Tentative Method of Test for Oxidation Resistance of Paraffin Wax (Peroxide Method); (2) revision and reversion to tentative of Standard Method of Test for Distillation of Gas Oil and Similar Distillate Fuel Oils (D 158 – 54); and (3) revision for immediate adoption of Standard Method of Test for Distillation of Gasoline, Naphtha, Kerosine, and Similar Petroleum Products (D 86 – 56).

Committee D-3 on Gaseous Fuels:

Report presented, in the absence of the chairman, by P. J. Smith and the following action taken:

Adopted as Standard, Revisions in:

Test for Vapor Content of Gaseous Fuels by Measurement of Dew-Point Temperature (D 1142 – 53)

Committee D-10 on Shipping Containers:

Report presented, in the absence of the chairman, by P. J. Smith and accepted as a report of progress.

Committee D-11 on Rubber and Rubber-like Materials:

Report presented by Simon Collier, chairman, and the following actions taken:

Accepted as Tentative:

Specs. and Methods of Test for Flexible Foams Made from Polymers or Copolymers of Vinyl Chloride (D 1565 – 58 T)
Tests for Flexible Urethane Foam (D 1464 – 58 T)
Defs. of Terms Relating to Rubber and Rubber-Like Materials (D 1566 – 58 T), revised by a change in title and deletion of definitions for "Rubber and Rubber-like," and "Vulcanization." Also in the definition of "International Rubber Hardness Degree" the last sentence

was omitted from the note. In the definition of "tear strength" the words "in pounds per inch of thickness" were omitted. In the definition of "tensile strength" the words "uniformly over the cross-section of the test specimen" were omitted. The definition of "tensile stress" was revised to read: "The applied force per unit of original cross-sectional area of a specimen." The definition of "tensile stress at given elongation" was revised to read: "The tensile stress required to stretch a uniform section of a specimen to a given elongation." In the definition of "test specimen" the word "physical" was omitted.

Accepted as Tentative, Revisions in:

Methods of Sample Preparation for Physical Testing of Rubber Products (D 15 – 57 T)
Methods of Testing Rubber-Coated Fabrics (D 751 – 57 T)
Specs. and Methods of Test for Latex Foam Rubbers (D 1055 – 58 T)
Test for Evaluating Pressure Sealing Properties of Rubber and Rubber-like Materials (D 1081 – 49 T)
Test for Harmful Dirt in Crude Natural Rubber (D 1278 – 53 T)
Methods for Chemical Analysis of Synthetic Elastomers (Solid Butadiene-Styrene Copolymers) (D 1416 – 58 T), with a number of editorial changes in the procedure for Determination of Mixed Alkylated Phenols (MAP) Content as preprinted.

There has been withdrawn from the Report of Committee D-11 the recommendation for the revision of Tentative Method of Test for Low-Temperature Brittleness of Rubber and Rubber-Like Materials (D 736 – 54 T) since the changes are of an editorial nature and had been previously incorporated in the method.

The revision of Tentative Method of Test for Brittleness Temperature of Plastics and Elastomers by Impact (D 746 – 55 T) has been withdrawn from the report since this standard, while of interest to Committee D-11, is under the jurisdiction of Committee D-20 on Plastics, and the revisions set forth in the D-11 Report constitute a recommendation to Committee D-20 and not to the Society.

Adopted as Standard:

Spec. for Natural Rubber AO Insulation for Wire and Cable (D 27 – 58 T)
Spec. for Natural Rubber Performance Insulation for Wire and Cable (D 353 – 58 T)
Spec. for Natural Rubber Heat-Resisting Insulation for Wire and Cable (D 469 – 58 T)
Spec. for Natural Rubber Sheath for Wire and Cable (D 532 – 54 T)
Spec. for Ozone-Resisting Insulation for Wire and Cable (D 574 – 54 T)
Spec. for Synthetic Rubber Insulation for Wire and Cable, 75 C Operation (D 754 – 58 T)
Spec. for Synthetic Rubber Insulation for Wire and Cable, 60 C Operation (D 755 – 58 T)
Spec. for GR-S Synthetic Rubber Sheath for Wire and Cable (D 866 – 54 T)
Test for Hardness of Rubber (D 314 – 52 T)
Test for Adhesion of Vulcanized Rubber to Metal (D 429 – 56 T)
Test for Compression Fatigue of Vulcanized Rubber (D 623 – 52 T)
Methods of Testing Adhesives for Brake Lining and Other Friction Materials (D 1205 – 53 T)

Adopted as Standard, Revisions in:

Test for Young's Modulus in Flexure of Natural and Synthetic Elastomers at Normal and Subnormal Temperatures (D 797 – 46)

Committee D-12 on Soaps and Other Detergents:

Report presented in the absence of the chairman by P. J. Smith, and the following actions taken:

Accepted as Tentative:

Method of Testing Detergent Cleaners for Evaluation of Corrosive Effects on Certain Porcelain Enamels (D 1567 – 58 T)
Methods for Sampling and Chemical Analysis of Alkylbenzene Sulfonates (D 1568 – 58 T)
Methods of Testing Detergent Alkylate (D 1569 – 58 T)
Methods for Sampling and Chemical Analysis of Fatty Alkyl Sulfates (D 1570 – 58 T)

Accepted as Tentative, Revisions in:

Def. of Terms Relating to Soaps and Other Detergents (D 459 – 57 T)
Methods for Sampling and Analysis of Sodium Triphosphate (D 501 – 56 T)

Adopted as Standard, Revisions in:

Methods of Sampling and Chemical Analysis of Soaps and Soap Products (D 460 – 54)
Methods of Sampling and Chemical Analysis of Alkaline Detergents (D 501 – 57)
Methods of Chemical Analysis of Industrial Metal Cleaning Compositions (D 800 – 45)
Methods of Chemical Analysis of Soaps Containing Synthetic Detergents (D 820 – 46)

Accepted for Publication as Information Only:

Test for Sodium Alkylbenzene Sulfonate in Synthetic Detergents by Ultraviolet Absorption

Committee D-15 on Engine Antifreezes:

Report presented by R. E. Vogel, chairman, and the following actions taken:

Accepted as Tentative, Revisions in:

Spec. for Hydrometer-Thermometer Field Tester for Engine Antifreezes (D 1124 – 53 T), further revised by retaining the present values for hydrometers in Table I and in paragraph (1) of Section 11(*b*).[28]

Adopted as Standard:

Test for Ash Content of Concentrated Engine Antifreezes (D 1119 – 53 T)
Test for pH of Concentrated Engine Antifreezes (D 1287 – 53 T)

Adopted as Standard, Revisions in:

Test for Specific Gravity of Concentrated Engine Antifreezes by the Hydrometer (D 1122 – 53)

Committee D-16 on Industrial Aromatic Hydrocarbons and Related Materials:

Report presented by D. F. Gould, chairman, and the following action taken:

Accepted as Tentative:

Method for Calculation of Volume and Weight of Benzene, Toluene, and Paraxylene (D 1555 – 58 T) (Jointly with Committee D-2)

Committee D-20 on Plastics:

Report presented by F. W. Reinhart, chairman, and the following actions taken:

Accepted as Tentative:

Abbreviations of Terms Relating to Plastics (D 1600 – 58 T), revised by the deletion in Section 2(*a*) of Poly(vinylidene chloride) .....PVC2, and the deletion in the Appendix of paragraph A5.
Spec. for Glass Fabric Reinforced Epoxy Resin Laminates (D 1592 – 58 T) (Jointly with Committee D-9)
Test for Dilute Solution Viscosity of Ethylene Polymers (D 1601 – 58 T)
Test for Bearing Load of Corrugated Plastics Panels (D 1602 – 58 T)
Test for Carbon Black in Ethylene Plastics (D 1603 – 58 T)
Method for Measurement of Flatness of Plastics Sheet or Tubing (D 1604 – 58.T)
Test for Time-to-Failure of Plastic Pipe Under Long-Term Hydrostatic Pressure (D 1598 – 58 T)
Test for Short-Time Rupture Strength of Thermoplastic Pipe, Tubing, and Fittings (D 1599 – 58 T), with editorial revisions, and revised by the addition of Section 7(*e*) covering fittings when tested alone and not in connection with a pipe specimen.
Spec. for Nonrigid Vinyl Chloride Plastic Sheeting (D 1593 – 58 T)
Spec. for Methylstyrene-Acrylonitrile Copolymer Molding and Extrusion Material (D 1594 – 58 T)
Spec. for Polymethylstyrene Molding and Extrusion Material (D 1595 – 58 T)

Accepted as Tentative, Revisions in:

Test for Tensile Properties of Plastics (D 638 – 56 T)
Nomenclature of Descriptive Terms Pertaining to Plastics (D 675 – 57 T)
Spec. for Cast Methacrylate Plastic Sheet, Rods, Tubes, and Shapes (D 702 – 56 T)
Def. of Terms Relating to Plastics (D 883 – 57 T), with the deletion of definitions for Urethane Plastics, Isotactic, and Forming.
Spec. for Polyethylene Molding and Extrusion Materials (D 1248 – 52 T)
Spec. and Methods of Test for Polychlorotrifluoroethylene Molding and Extrusion Materials (D 1430 – 56 T)

Adopted as Standard:

Test for Flammability of Flexible Thin Plastic Sheeting (D 1433 – 56 T)
Test for Gas Transmission Rate of Plastic Sheeting (D 1434 – 56 T)
Rec. Practice for Outdoor Weathering of Plastics (D 1435 – 56 T)
Method of Sampling and Testing Plasticizers Used in Plastics (D 1045 – 55 T)
Spec. for Nonrigid Poly(Vinyl Chloride) and Copolymer Molding and Extrusion Compounds (D 1432 – 56 T)

Accepted for Publication as Information Only:

Test for Measuring the Surface Flammability of Plastics Using a Radiant Heat Energy Source

The committee withdrew from the report as preprinted the following recommendations: (1) Proposed new Tentative Method of Test for Impact Resistance of Thin Plastic Sheet by Free Falling Dart Method; and (2) Proposed new Tentative Spec. for Woven Glass Fabric, Cleaned and Finished for Plastic Laminates.

The revision of the Method of Test for Deflection Temperature of Plastics Under Load (D 648 – 56), set forth in the preprinted report under Revision of Tentatives, should have been indicated as an editorial change.

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[28] Subsequent to the Annual Meeting, as a result of action of Committee E-1 with respect to ASTM Specifications for Hydrometers (E 100 – 57 T) an editorial revision was made changing the values for hydrometers to read as originally preprinted.

FORTY-FIRST SESSION—COMMITTEE REPORT SESSION

FRIDAY, JUNE 27, 12:30 P.M.

SESSION CHAIRMAN: D. E. PARSONS

Committee C-1 on Cement:

Report presented by R. R. Litehiser, chairman, and the following actions taken:

Accepted as Tentative, Revisions in:

Methods of Chemical Analysis of Portland Cement (C 114 – 57 T)
Methods of Sampling Hydraulic Cement (C 183 – 55 T)
Spec. for Portland Blast-Furnace Slag Cement (C 205 – 57 T)
Spec. for Air-Entraining Additions for Use in the Manufacture of Air-Entraining Portland Cement (C 226 – 54 T)
Spec. for Portland-Pozzolan Cement (C 340 – 57 T)
Test for Air Content of Hydraulic Cement Mortar (C 185 – 55 T)[29]
Test for Potential Alkali Reactivity of Cement-Aggregate Combinations (C 227 – 52 T)[29]
Test for Bleeding of Cement Pastes and Mortars (C 243 – 57 T)[29]
Test for Calcium Sulfate in Hydrated Portland Cement Mortar (C 265 – 57 T)[29]
Test for Time of Setting of Hydraulic Cement by Gillmore Needles (C 266 – 51 T)[29]
Method for Mechanical Mixing of Hydraulic Cement Mortars of Plastic Consistency (C 305 – 55 T)[29]

Adopted as Standard:

Spec. for Slag Cement (C 358 – 57 T)

Adopted as Standard, Revisions in:

Spec. for Masonry Cement (C 91 – 57)
Test for Compressive Strength of Hydraulic Cement Mortars (Using 2-in. Cube Specimens) (C 109 – 56)[29]
Methods of Chemical Analysis of Portland Cement (C 114 – 53)
Test for Fineness of Portland Cement by the Turbidimeter (C 115 – 53)
Test for Autoclave Expansion of Portland Cement (C 151 – 56)[29]
Test for Normal Consistency of Hydraulic Cement (C 187 – 55)[29]
Test for Tensile Strength of Hydraulic Cement Mortars (C 190 – 49)[29]
Test for Time of Setting of Hydraulic Cement by Vicat Needle (C 191 – 52)[29]

Committee C-11 on Gypsum:

Report presented in the absence of the chairman by R. H. Faber, and the following actions taken:

Accepted as Tentative, Revisions in:

Spec. for Precast Reinforced Gypsum Slabs (C 377 – 56 T)
Spec. for Annular Ringed Nails for Gypsum Wallboard (C 380 – 56 T)

Adopted as Standard, Revisions in:

Spec. for Gypsum Plasters (C 28 – 55)
Spec. for Gypsum Wallboard (C 36 – 55)
Def. of Terms Relating to Gypsum (C 11 – 55)

The revisions for immediate adoption set forth in the preprinted report of Standard Specifications C 317 – 55 and Standard Methods C 26 – 56 comprise editorial changes which were made in the standards in 1957. Accordingly, these revisions will be deleted from the report.

Committee C-13 on Concrete Pipe:

Report presented by R. R. Litehiser, chairman, and the following actions taken:

Accepted as Tentative, Revisions in:

Spec. for Concrete Pipe for Irrigation or Drainage (C 118 – 56)

Adopted as Standard, Revisions in:

Spec. for Concrete Sewer Pipe (C 14 – 57)

Committee C-14 on Glass and Glass Products:

Report presented in the absence of the chairman by R. E. Hess, and accepted as a report of progress.

Committee C-15 on Manufactured Masonry Units:

Report presented by J. W. Whittemore, chairman, and the following actions taken:

Adopted as Standard, Revisions in:

Spec. for Sewer Brick (Made from Clay or Shale) (C 32 – 50)
Spec. for Building Brick (Solid Masonry Units Made from Clay or Shale) (C 62 – 57)

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[29] The general recommendation concerning the addition of a requirement for relative humidity of a laboratory applies to this method.

Committee C-16 on Thermal Insulating Materials:

Report presented in the absence of the chairman by R. E. Hess, and the following actions taken:

Accepted as Tentative:

Test for Mechanical Stability of Preformed Thermal Insulation by Tumbling (C 421 – 58 T)
Method of Making and Curing Test Specimens of Mastic Thermal Insulation Coatings (C 419 – 58 T)
Test for Thermal Conductivity of Insulating Materials at Low Temperatures by Means of the Wilkes Calorimeter (C 420 – 58 T)

Adopted as Standard:

Spec. for Molded Cork Pipe Insulation for Low Temperatures (C 339 – 55 T)
Spec. for Cellular Glass Thermal Insulation for Pipes (C 381 – 56 T)
Test for Breaking Strength and Calculated Flexural Strength of Preformed Block-Type Thermal Insulation (C 203 – 55 T)
Test for Wet Adhesion of Thermal Insulating Cements to Metal (C 383 – 56 T)

Committee D-8 on Bituminous Waterproofing and Roofing Materials:

Report presented by H. R. Snoke, chairman, and the following actions taken:

Accepted as Tentative, Revisions in:

Methods of Sampling and Testing Felted and Woven Fabrics Saturated with Bituminous Substances for Use in Waterproofing and Roofing (D 146 – 56 T)
Methods of Testing Asphalt-Base Emulsions for Use as Protective Coatings for Built-Up Roofs (D 1167 – 51 T)
Spec. for Asphalt Insulating Siding Surfaced with Mineral Granules (D 1226 – 52 T)
Spec. for Asphalt Shingles Surfaced with Mineral Granules (D 225 – 54)
Spec. for Asphalt Roll Roofing Surfaced with Mineral Granules (D 249 – 56)
Test for Coarse Particles in Mixtures of Asphalt and Mineral Matter (D 313 – 41)

Adopted as Standard:

Spec. for Wide Selvage Asphalt Roll Roofing Surfaced with Mineral Granules (D 371 – 55 T)
Methods of Testing Asphalt Insulating Siding Surfaced with Mineral Granules (D 1228 – 52 T)
Test for Staining Properties of Asphalts (Modified Pressure Method) (D 1328 – 54 T)
Test for Contact Compatibility Between Asphaltic Materials (Oliensis Test) (D 1370 – 55 T)
Methods of Testing Asphalt Emulsions for Use as Protective Coatings for Metal (D 1010 – 55 T) (Jointly with Committee D-1)

Adopted as Standard, Revisions in:

Spec. for Asphalt Roll Roofing Surfaced with Powdered Talc or Mica (D 224 – 55)
Spec. for Asphalt Shingles Surfaced with Mineral Granules (D 225 – 54)
Spec. for Asphalt Siding Surfaced with Mineral Granules (D 699 – 54)

Standard Withdrawn:

Spec. for Coal-Tar Pitch for Steep Built-Up Roofs (D 654 – 49)

FORTY-SECOND SESSION—SYMPOSIUM ON APPLICATION OF SOIL TESTING IN HIGHWAY DESIGN AND CONSTRUCTION[26]

(*Symposium Continued from the Thirty-seventh Session*)

FRIDAY, JUNE 27, 2:00 P.M.

SESSION CHAIRMAN: E. J. KILCAWLEY

Committee D-18 on Soils for Engineering Purposes:

Report presented by E. J. Kilcawley, chairman, and the following actions taken:

Accepted as Tentative:

Test for Density of Soil in Place by the Sand Cone Method (D 1556 – 58 T) (Jointly with Committee D-4)
Test for Moisture-Density Relations of Soils Using a 10-lb Rammer and an 18-in. Drop (D 1557 – 58 T) (Jointly with Committee D-4)
Test for Moisture-Penetration Resistance Relations of Fine-Grained Soils (D 1558 – 58 T) (Jointly with Committee D-4)
Methods for Thin-Walled Tube Sampling of Soils (D 1587 – 58 T)
Method for Penetration Test and Split-Barrel Sampling of Soils (D 1586 – 58 T)

Accepted as Tentative, Revisions in:

Test for Moisture-Density Relations of Soils (D 698 – 57 T) (Jointly with Committee D-4)

Adopted as Standard, Revisions in:

Method of Preparing Soil Samples for Grain-Size Analysis and Determination of Subgrade Soil Constants (D 421 – 39)
Test for Specific Gravity of Soils (D 854 – 52)

Symposium Papers:

Laboratory Methods of Compacting Granular Materials—E. J. Felt, presented by the author.
Ohio's Typical Moisture-Density Curves—J. G. Joslin, presented by the author.
Soil Classification Scheme Based on Compaction Tests—J. L. McRae, presented by the author.
Laboratory Shear Tests on Varved Clays and Their Application to Design—R. E. Machol (presented by title only).
The Value of Soil Test Data in Local and Regional Road Planning—Martin Ekse, summary presented by H. B. Seed.

EDITOR'S NOTE.—The recommendations affecting standards referred to letter ballot were approved when the ballot was canvassed on September 22.

A CONTINUING AND VITAL CHALLENGE

ANNUAL ADDRESS BY THE PRESIDENT

RICHARD T. KROPF[1]

JUNE 24, 1958

Since the very beginning of history, man has faced the future with great courage and with feelings of deep concern over the dark unknown which lay ahead. It was with this great courage that ancient man and all of those who have gone before us met and mastered the great challenges of their unknown future. It is to them we are indebted for our great heritage of today. However, with this priceless inheritance we have also fallen heir to the great responsibility of meeting and mastering the great challenges of our future.

You may well say that ours is a far greater responsibility today than those of yesteryear, with our ever-accelerating progress. Let us not, however, permit the magnitude of the absolute values to obscure the relative factors involved. Leave for a moment the consideration of our problems of the future and, in the "time machine" of your imagination, transport yourself from the comfort and security of your present surroundings, back twenty-five thousand years to a damp, dark cave in what is now Dordogne, France.

IN THE BEGINNING

Clad only in a tattered pelt, the shrill wintery blasts outside the cave chill you to the bone, and the howls of the wild animals stalking you chill the very marrow of these already well-chilled bones. You are cold; you are hungry; you may be desperately ill. You are superstitious; you are afraid; you are worried. Worried about the "future?" Hardly, since you are worried about your next meal; you are worried that you might not live to eat that meal even if you knew from whence it might come. Would you trade our responsibilities of today for those of primitive man? I am certain that no one among us would entertain such an idea—for over the past 250 centuries our progress has been great and the benefits which have accrued to mankind are virtually beyond measure.

Let us pause for a moment with our cave men to witness that which historians consider to be the first major step in the history of cilivization. We find our primitive man using simple stones as tools and as weapons for killing animals and fighting his enemies. Several of these stones he treasures above all others because they are of a more usable shape and have a somewhat sharp edge, and he eagerly seeks more of them. Then, through his primitive means of communicating ideas, he learns that there is available in a strange and distant place—actually relatively but a short distance by our standards—a marvelous NEW MATERIAL which can be shaped into better, more efficient, sharper tools and more effective weapons. This material was not easy to obtain and must meet certain requirements.

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[1] Vice-President and Director of Research, Belding Heminway Co., Inc., New York, N. Y.

EARLIEST RESEARCH AND STANDARDS

It is of particular interest to us of the American Society for Testing Materials, which is dedicated to "research and standards for materials," that we are observing, as primitive as it may be, the very earliest "research and standards for materials." Now, what was this marvelous new history-making material? It was flint! Not the common variety that had lain on the surface of the earth, eroding for countless centuries, but the kind which was dug from beneath the surface. Primitive research had shown that this flint could be "flaked" so that it could be worked into various shapes with sharp edges. This must have been called "high-test flint," and before our cave man would accept it, he tested it for "flaking" by striking together two pieces, one held in each hand. Here we observe the earliest accepted "test method and standard for material." Yet, as intriguing as this may be because of our specific technological interests, we must not lose sight of the significance of this to the progress of civilization.

MORE NEW HORIZONS

To mankind this was not only a new material—it was a NEW HORIZON. Historians tell us that this may be considered the primary stimulus of our civilization. Our cave man now traveled many miles to the flint mines of northern Europe, some of which in existence today are reported to be over 17,000 years old. Ancient man traded furs and amber for this "high-test flint," and it is generally agreed that this is the very first evidence of commerce or trade. This stimulated the development of various means of the better communication of ideas, and certain archeologists claim that it can be shown that there were even traveling flint salesmen at that time.

The centuries moved slowly, and the next step in progress was to be marked by a new material and a NEW HORIZON with the discovery of copper. Progress continued to move at a faster pace and we arrive at another NEW HORIZON, on which we find a new material—bronze! Associated with this "royal metal," as it was often described, the horizon of civilization expanded rapidly to still another NEW HORIZON. Here we find another new material —iron; and with this material the horizons of civilized man have extended almost beyond our comprehension.

As we have made this rapid passage through the pages of history there is one fact that stands out above all others. The history and progress of civilization are inseparable from materials. This has long been recognized by historians who have described these periods of our history by the materials we saw on each NEW HORIZON: the Stone Age, the Copper Age, the Bronze Age, and our own Iron Age.

MATERIALS USE—A YARDSTICK

It is quite logical, therefore, that material usage may be considered a yardstick of the progress of civilization, and in order that we may picture this growth let us consider the usage of iron in our period of this Iron Age. In the middle of the last century about 175 lb of iron were consumed per capita per annum in the highly industrialized countries. By the turn of the century, we were using approximately 440 lb; and by 1950 we were consuming about 1000 lb per capita per annum. We entered 1957 with a rate of approximately 1300 lb, which means we have been doubling our consumption of iron approximately every thirty-five years. An even more dramatic measure of our progress is recognized in the use of electrical energy. Here we find that we double our use of electricity approximately every ten years!

Thus we may contemplate our future with some quantitative estimate of the magnitude of the challenge which lies ahead. It is but necessary to consider the growth rate of our population and compound this with our accelerating usage rates of materials and energy to develop an array of astronomical numbers which represent the requirements of our civilization by the year 2000. Time will not permit a recapitulation of these data; however, they may be dramatically translated into terms of the *scientific and engineering brainpower* required to meet this *great challenge* less than forty-two years hence. This era of exploding technology will require, by the year 2000, more than ten times the number of scientists and engineers we have among us today; and, what is far more serious, this is roughly three times the number which will be produced by our educational system at our current rate of production. This situation has deeply concerned thinking men and women in every walk of life, and enlightened people everywhere have put their shoulder to the wheel in an effort to solve this vital problem.

ASTM's GREAT RESPONSIBILITY

You can be very proud of the important role which you individually and collectively through our great American Society for Testing Materials are playing and are destined to play in this important program. To understand fully the importance of ASTM and its position of responsibility in America and the entire civilized world, it is important to recognize two fundamental facts: First, materials are the common denominator of all engineering; and, second, the ASTM must be correctly described as the "American Society for Materials." Thus, ours is the sacred trusteeship of that vital factor—materials—common to all engineering, and essential to all progress.

The unreserved support and respect given the ASTM by the individual members of every branch of the scientific and engineering professions, by the many educational and research institutions of the nation, by our ever-growing industry, by the many agencies of our various local, state, and federal governments, and above all by nontechnical people of very walk of life, is mute evidence of the recognition of the important role it has played and is destined to play in our future. It is not my intention to recapitulate here the details of the outstanding contribution which the ASTM has made and is continuing to make, since this is well documented in, and by, our many publications and our monumental Book of Standards. It should be pointed out, however, that this support and respect has been earned by the ASTM through the responsibilities which it has assumed and discharged with distinction, through the dedicated efforts of its members, its myriad committees, and its outstanding staff.

Proud as we may be of our accomplishments, our *great challenge* still stands before us. On every NEW HORIZON of every new day, we can truly say there is a new material with the need for new and more research and standards which is the fundamental responsibility of ASTM. Coupled with this is the responsibility of the more effective communication of the information related to this common denominator of all engineering to all engineers and scientists and thereby multiply their effectiveness and thus compensate in part for the anticipated serious shortage in their numbers. Ours is the responsibility to stimulate and to assist in the field of scientific and engineering education, particularly in those areas related to materials. These are but a few of our responsibilities which will continue to grow and to change with the passing of time.

WORKING FOR THE FUTURE

As you are aware, your Society is deeply conscious of these challenges and responsibilities and has currently underway extensive programs on long-range planning, education, and other matters; to mention but one, the anticipated mushrooming demand for publication growth through this exploding technology. Each member and each committee is playing a vital part in this program, and it is with pride that we can say that ASTM is a working Society and that it is working for the future!

Likewise, it is unnecessary for me to say, I am sure, that I share your enthusiasm and confidence in the ASTM and in its ability to meet every *great challenge* on every *new horizon*, since my thoughts of our distinguished Society, its members and its future, were well expressed by Henry David Thoreau, the Sage of Walden Pond, when he said in "What I Lived For":

"To him whose elastic and vigorous thought keeps pace with the sun the day is a perpetual morning."

1958 ANNUAL REPORT OF THE BOARD OF DIRECTORS

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1957–1958 was the period of sputniks, guided missiles, jet aircraft, and basically the period when perhaps for the first time there was mass realization of the significance of scientific training and scientific research.

What did ASTM do in this period? Did it progress or remain static?

With all the emphasis on science there was also an increasing realization of the significance of materials. In many fields the cries were for newer and better materials to stand higher temperatures and heavier stresses.

But this is not a new situation. Ever since ASTM came into being in 1898, each year has seen industry and government bringing new problems to the Society for study. Many of the answers can be obtained only through cooperative work, and the whole philosophy of our national technical Society is based on this premise—cooperation between the producer and consumer, between government and industry, between professional and technical societies.

The tremendous breadth of ASTM work involving so many different materials makes it impossible to summarize in this Directors' Report even the new work started for the first time, let alone the completion of many other research and standards projects previously initiated. The reports of the some 80 main technical committees comprising some 600–800 pages as preprinted, and the ASTM BULLETIN are the only means of gaining a comprehensive picture of what is transpiring. *However*, some major new activities are noted in the succeeding section entitled "Technical Activities."

There are many outstanding examples where cooperative work between different industries and associations has made remarkable developments—for example, the electric power field started on a commercial basis 75 years ago last September 4 when the Pearl Street Edison Central Station went into operation. What a far cry from that small but significant project to those now on the drawing boards or under construction with operating temperatures contemplated of 1200 F or greater and pressure of 5000 psi. Many have contributed to make these new 300,000- or 400,000-kw turbine generators possible, including several ASTM committees which through research and standards have contributed to the production and use of better materials.

It is pleasant to contemplate accomplishments, but what of the future?

10-Year Planning Committee:

What kind of service will industry and Government be expecting from ASTM in 1968? Is our present organization sufficient to meet the needs?

Should ASTM standardization work be pushed more actively? Is the Society's name a misnomer considering that it is not a Society for Testing but the outstanding technical organization for materials technology?

These, a part of some 60 penetrating questions, are now being studied by a Long-Range Planning Committee appointed by the Board of Directors. Other officers and members are helping and more will be asked to participate.

The objective is to anticipate as best we can how the Society should be set up to give the services expected. It is felt a look ahead of about 10 years is all that can be reasonably expected. Some recommendations from the Long-Range Planning Committee have already been approved by the Board (see later pages of this report), and more will be forthcoming.

In the midst of these studies the Society is hard pressed to keep abreast of the steady demands made on it, the very extensive and expanding publication schedule, and coordination of the many technical activities.

As one steps aside from the intensive activities he is impressed by the tre-

HIGH LIGHTS, 1957 FINANCIAL OPERATIONS

	1957	1956	1955
Gross Receipts:			
Membership Dues	$283 400	$269 000	$252 100
Sales of Publications	623 900	695 950	521 500
Other	113 400	117 600	78 200
Total	$1 020 700	$1 082 550	$851 800
Expenditures:			
Salaries	$360 200	$309 400	$305 400
Printing	430 200	342 300	350 000
Other	237 800	244 800	196 000
Total	$1 028 200	$896 500	$851 400
Favorable Operating Balance	...	$186 050	$400
Operating Deficit	$7 500	...	...
Net Assets:			
Real Estate	$271 500	$269 450	$277 700
Investments and Cash	1 251 300	$1 048 315	880 500
Equipment	36 900	32 900	29 500
Total	$1 559 700	$1 350 665	$1 187 700
Asset Equity of Each Member	$170	$160	$147
Number of Members	9 159	8 408	8 079
	1957	1956	1955
Pages of Publications	10 276	10 540	18 742
Number of Employees	78	75	69

mendous outpouring of service—technical skills, research facilities, financial backing—which the Society receives from its members, committee members, and friends. This great aggregate marshalled under the Board and its technical committees is responsible for the tremendous outpouring of technical services the Society has been so gratified to make.

With the increasing tempo of technical progress—and exploding technology—we must continue to count on these and additional sources of help in order to meet the challenges.

Technical Activities

THE technical committees, of which there are some 80 main committees and hundreds of subcommittees and sections, are really the "heart" of ASTM. Their annual reports as preprinted, covering important research and standards activities, total some 600 to 800 pages. Members receive preprints on request and thus are currently posted on major work of interest. In this Directors' Report, reference is made only to significant changes in committee scopes and to important major new work.

Revised Committee Scopes and Jurisdictions:

Committee D-8 has received authorization of the Board to include bituminized fiber pipe in its activities. The title of the committee is therefore being changed from Committee D-8 on Bituminous Waterproofing and Roofing Materials to "Committee D-8 on Bituminous Roofing, Waterproofing, and Related Building and Industrial Materials," and the scope is being revised to read as follows by the deletion of the words in brackets and the addition of the words in italics:

> "The formulation of specifications, methods of test, and definitions of terms pertaining to bituminous [waterproofing, roofing and siding] materials *and products for building and industrial use.*"

The scope of Committee E-13 on Absorption Spectroscopy is being changed from its present form, namely,

> "The coordination and formulation of methods of spectroscopic analysis by absorption, fluorescence and scattering techniques,"

to read:

> "The advancement of the fields of spectroscopy involving molecular, atomic, or nuclear absorption, and fluorescence, scattering, or polarization, by promoting exchange of information, by sponsoring meetings and symposia for presentation of papers, by standardizing nomenclature relating to absorption spectroscopy, and by coordinating and formulating scientific practices and methods of analysis in these fields."

Major New or Suggested Activities:

Constantly there are brought to the Society suggestions for standardization or research work in fields which have not previously been covered. These suggestions are always welcome and the Board of Directors gives careful consideration to them. Usually, following preliminary discussions with leaders in the field, representing both consumer and producer, a conference will be called with rather widespread representation. This group may then make specific recommendations or ask that further information be obtained before reaching conclusions.

It should be remembered too that constantly the existing main technical committees of the Society are undertaking new work for new products and materials which are covered in their scopes. Thus each year there are from 75 to 100 or more new standards issued covering important tests or specification requirements. These are publicized in the ASTM BULLETIN, in the annual reports of the committees, and in the technical press.

Certain new activities of a major activity are noted here:

Flexible Barrier Material.—A conference of interested persons, held at Society Headquarters on May 28, 1957, recommended that a new technical committee be established. A steering committee organized at the conference held a meeting and agreed upon three types of materials for possible consideration, namely, basic materials, combined or composite materials, and barrier applications. Following approval by the Board, the committee held an organization meeting on December 12, 1957, and recommended the following scope which has received Board approval:

> "The development of definitions of terms and nomenclature, methods of test and specifications for flexible barriers, including basic and composite materials and their applications, and the promotion of research in this field. Standards covered by other ASTM committees shall be used when applicable."

The committee has been designated "Committee F-2 on Flexible Barrier Materials." (The F series are "use" committees in areas where one or more basic materials may be involved.)

Halogenated Organic Solvents and Solvent Mixtures.—A new ASTM committee on Halogenated Organic Solvents and Solvent Mixtures was recommended at a December, 1957, conference attended by a large group of consumers and producers. It was originally visualized as a use committee but a materials committee was recommended covering halogenated solvents, whatever their use, except for certain solvent requirements now under the jurisdiction of Committee D-1 on Paint, Varnish, Lacquer, and Related Products and solvent materials now under the jurisdiction of Committee D-2 on Petroleum Products and Lubricants. The Board approved the formation of this new committee. The tentative scope for this proposed new committee, worded so as to avoid overlap and duplication with other ASTM committees concerned with solvents, is set forth below:

> "The promotion of knowledge pertaining to halogenated organic solvents and admixtures thereof including formulation of specifications, definitions, and methods of test."

The committee is holding its organization meeting during the ASTM Annual Meeting in Boston. A steering committee under the chairmanship of M. A. Pinney of the Pennsylvania Railroad Co., a member of the ASTM Administrative Committee on Standards, is planning the organization.

Cellular Materials.—Although some work has been carried on in certain of the technical committees covering cellular materials, several existing committees are concerned with various aspects of these materials. To avoid duplication of effort and to channel some of the activities, a coordinating committee was appointed several years ago, but recently Committee D-20 on Plastics recommended that a new technical committee be established and that it be given jurisdiction of work which should be under way or under consideration in six existing main committees. The Board has referred this suggestion to the coordinating committee for resolution, if possible, and advice.

Industrial Chemicals.—A study and advisory committee on Industrial Chemicals was appointed by the Board to consider various recommendations from earlier conferences and in general to advise the Board on new work in this field which would be constructive and receive the support of producer and consumer. This group, headed by W. A. Kirklin of Hercules Powder Co., has held several meetings and its latest recommendation, part of an extensive report, is that there be formed a new "E" committee to be responsible for test methods and related standards applicable to the broad field or families of such chemicals.

The title proposed for the new committee is "Committee E-15 on Analysis and Testing of Industrial Chemicals"; the proposed scope is as follows:

(a) To develop or formulate standard methods for the analysis and testing of industrial chemicals.[a]

NOTE.—Specifications are not included within the scope of this committee.

(*b*) In cooperation with other committees of the Society, to coordinate and reconcile chemical analytical methods for nonmetallic materials and chemicals that are broadly applicable and, where appropriate, to develop or formulate such methods.

(*c*) To encourage the use of uniform methods of analysis.

(*d*) To formulate standards for nomenclature, definitions and methods of sampling pertaining to analysis and testing of industrial chemicals and chemical analysis of nonmetallic materials.

(*e*) To establish precision and accuracy of standard analytical methods and test methods for chemicals.

(*f*) To encourage the establishment of new product committees in the field of industrial chemicals where needs become evident.

Coated Abrasives and Abrasive Grain.—The subject of standards on abrasives has been considered for some time. There are two principal areas, the coated abrasive field and abrasive grains *per se.* Discussions have been held with representatives of associations and companies in the field, and while no new committee has yet been recommended, a proposed Method of Test for Bulk Density of Abrasive Grain, prepared by the Abrasive Grain Assn., is being referred to Committee E-1 on Methods of Testing for processing.

Cermets.—The Board of Directors appointed a study committee under the temporary chairmanship of Past-President N. L. Mochel, which met in St. Louis during ASTM Committee Week and elected J. R. Tinklepaugh of Alfred University as permanent chairman. The members agreed to review existing ASTM and other standards as to their adaptability to use with cermets, with or without modification. Further meetings of the study committee are to be held. Whether a new technical committee will be recommended depends on the outcome of these meetings. It would seem that some constructive work involving both research and standards could be carried out under Society auspices.

Bulk Sampling.—Committee E-11 on Quality Control is considering means to implement and keep constant pressure behind recognized sampling or bulk sampling methods. This may require engaging a competent technical individual to work solely in this field under Committee E-11 or some joint auspices. A symposium on bulk sampling is being held at the Annual Meeting in Boston.

Gaskets.—At a conference on May 1 in Philadelphia with over 70 persons present representing producers and consumers, other technical societies, and the U. S. Government, all concerned with this field, it was decided to appoint a study committee to develop proposals of how ASTM could render further service in this field. Committee D-11 on Rubber and Rubber-Like Materials has had an active subcommittee for many years with several standards resulting. There are essentially four broad areas to be considered, namely, (1) material tests and specifications, (2) design considerations, (3) performance specifications, and (4) dimensions and related problems. This group, which is to meet in the near future, is expected to make recommendations as how best the ASTM can function in this field.

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[a] Industrial chemicals as used in this scope are defined as follows: "Single elements or compounds in various stages of purity for consumption primarily by the chemical industry for further conversion."

National and Other Meetings

IN Research and Standards for Materials, as in many other fields, it is essential the participants meet occasionally to share information, review progress, and plan for the future.

1957 Annual Meeting:

The Annual Meeting in Atlantic City, June 16–21, 1957, the Society's Sixtieth, included more than 800 meetings of technical committees with as many as 60 running simultaneously, some 30 technical sessions, and numerous special luncheons and dinners. All of the committee reports and a number of the technical papers appear in the 1957 *Proceedings*, and numerous technical papers organized as symposiums or special sessions have been issued as separate special technical publications (STP's). Everyone at the meetings benefits from the technical and committee programs. There was a review of accomplishments, and plans were made for the year ahead. A condensed account of the meeting, but still covering 32 pages, is featured in the July 1957 ASTM BULLETIN.

1958 Committee Week:

For the first time Committee Week was held west of the Mississippi—in St. Louis throughout the week of February 10. This period provides committees another opportunity to discuss and work on their problems. The coordinated committee program has been found to be more convenient for the hundreds of committee members who serve on different groups which otherwise might meet at different places at different times. There were about 280 committee meetings during this week.

As indicated under district activities, the Committee Week was featured by two industry luncheons—one on steel, the other, chemicals—and an outstanding dinner.

District Cooperation and National Meetings:

One of the very effective ways in which ASTM districts help the Society is by serving as a nucleus for local committees on arrangements for national meetings. The Philadelphia District consistently has sponsored ladies' entertainment and annual banquets at the Atlantic City Meetings, and the St. Louis District, through a general committee, deserves much credit for the outstanding success of the 1958 Committee Week special features. The 1958 Annual Meeting that is to be held in Boston throughout the week beginning June 22 has a very strong technical program. There is to be an extensive apparatus exhibit, and again hundreds of committees will meet. The General Committee on Arrangements formed by the New England District has many special events planned for the ladies and others. In addition to the President's Annual Luncheon, four special industry luncheons are being held covering Road Materials, Petroleum, Copper and Brass, and Instruments and Apparatus Industries.

Future Meetings:

Annual Meetings of the Society have been scheduled as follows:

June 22–27, 1958—Boston, Mass.
June 21–26, 1959—Atlantic City, N. J.
June 26–July 1, 1960—Atlantic City, N. J.
June 25–30, 1961—Atlantic City, N. J.
June 24–29, 1962—New York, N. Y.
June 23–28, 1963—Atlantic City, N. J.

Committee Week and Spring Meetings:

February 2–7, 1959—Pittsburgh, Pa.
February 1–5, 1960—Chicago, Ill.
January 29–February 3, 1961—Cincinnati, Ohio
February 5–9, 1962—Dallas, Tex.
February 3–8, 1963—Montreal, Quebec, Canada

Pacific Area National Meetings:

October 11–16, 1959—San Francisco, Calif.
September 30–October 5, 1962—Los Angeles, Calif.

District and Related Activities

WITH our membership coming from all professional fields, local activities are not handled through chapters but through fifteen ASTM districts operated by councils which in various ways effectively promote ASTM work in their areas. For many members district activities provide a means of meeting national and local ASTM officers.

During the year the activities of local Districts continued to occupy an important role in advancing the work of the Society. A new District, the Southeastern, was established, recognizing the industrial advances in this section of the country. A new Society District in Central New York is in the process of organization as is a new District in the Rocky Mountain area.

The Student Membership Awards Program has reached a new high with over 450 presentations made this year. Nine Districts have active programs. Three Districts have programs under study and one will renew its program next year.

President Kropf, in keeping with the pattern set by past presidents, toured the nation and spoke at many District and related meetings. E. P. Partridge, the 1957 Marburg lecturer, addressed two Districts and Alvin J. Herzig, 1957 Gillett Memorial lecturer, addressed three Districts, as indicated in the accompanying chart. A one-day meeting on "Life Testing" was held in Albuquerque, N. Mex., at which time plans for the Rocky Mountain District were also formulated.

The accompanying chart shows that 29 District meetings were held this year, several with very high attendance. The majority of the meetings were jointly sponsored with other technical and professional groups. News accounts of these meetings carrying speakers, subjects and cooperating groups and institutions appeared in various issues of the ASTM BULLETIN.

District Meetings:

New York District	September 26, 1957	New York, N. Y.
St. Louis District (jointly with AWS)	October 10, 1957	St. Louis, Mo.
New England District	October 31, 1957	Cambridge, Mass.
Southwest District (jointly with Tex. Soc. of Prof. Engrs., AIChE, ACS, NACE, ASCE)	November 11, 1957	Houston, Tex.
Western New York District	November 11, 1957	Buffalo, N. Y.
Pittsburgh District (jointly with Amer. Ceramic Soc.)	November 12, 1957	Pittsburgh, Pa.
Ohio Valley District (jointly with ACS, ASM)	November 12, 1957	Columbus, Ohio
Detroit District	November 13, 1957	Detroit, Mich.
Northern Calif. District (jointly with National Assn. of Power Engrs.)	November 15, 1957	San Francisco, Calif.
New York District	November 19, 1957	New York, N. Y.
Southwest District (with So. Regional Res. Lab., U. S. Dept. of Agric.)	November 19, 1957	New Orleans, La.
Southeastern District	November 20, 1957	Columbus, Ga. and Shawmut, Ala.
Washington District	November 21, 1957	Raleigh, N. C.
Philadelphia District	November 25, 1957	Philadelphia, Pa.
Southern Calif. District (jointly with ASM)	January 23, 1958	Los Angeles, Calif.
New Mexico (Special meeting)	January 27, 1958	University of New Mexico, Albuquerque, N. Mex.
Northern Calif. District (jointly with ASME)	January 30, 1958	San Francisco, Calif.
Southwest District (jointly with ASTM Committee D-2)	February 3, 1958	Houston, Tex.
New York District (with N. Y. Paint & Varnish Club)	February 6, 1958	New York, N. Y.
Philadelphia District	February 18, 1958	Philadelphia, Pa.
Ohio Valley District (jointly with ASTM Committee D-1)	March 6, 1958	Louisville, Ky.
Chicago District (with 1958 Nuclear Congress)	March 19, 1958	Chicago, Ill.
Washington District (jointly with ASTM Committee D-13)	March 20, 1958	Washington, D. C.
Cleveland District (jointly with ASM)	April 1, 1958	Cleveland, Ohio
Detroit District	April 21, 1958	Detroit, Mich.
Pittsburgh District	April 30, 1958	Pittsburgh, Pa.
Western New York-Ontario District	May 16, 1958	Buffalo, N. Y.
St. Louis (jointly with Washington Univ.)	May 17, 1958	St. Louis, Mo.
Philadelphia District (jointly with ASQC)	May 21, 1958	Trenton, N. J.

The St. Louis District organized a large General Committee to be host to the Committee Week meeting in February. Two industry luncheons were held, in the fields of Steel and Chemicals, with addresses respectively by N. P. Veeder, President, Granite City Steel Co., and Carroll A. Hochwalt, Vice-President, Monsanto Chemical Co. At the St. Louis Dinner, William M. Holaday, a member of the Society, and Director of Guided Missiles, Department of Defense, was the featured speaker. Many of St. Louis' industrial leaders were at the head table or in the audience.

The Philadelphia District acted as hosts for the 1957 Annual Meeting, sponsoring the ladies' entertainment and the entertainment following the annual banquet. The New England District will handle similar arrangements for the 1958 meeting in Boston.

In addition to technical sessions, the Districts have played an active role in local technical councils, science fairs, and the Society's Advisory Committee on Corrosion.

The Administrative Committee on District Activities held a meeting during the 1957 Annual Meeting and sponsored a breakfast to which District Council Officers were invited. Once again, this proved an excellent opportunity for the exchange of ideas relating to the promotion of the Society and its activities.

Revisions in ASTM By-Laws

Recommendations to Increase the Number of Directors; Raise Company and Sustaining Members' Dues:

Based on suggestions from the Long-Range Planning Committee, the Board of Directors has carefully considered two proposals—one to increase the number of Directors from fifteen to eighteen, and the other to raise the dues for Company and Sustaining Members. The Board recommends to the members that appropriate revisions in the By-laws be made to effectuate these changes.

The reason for a larger Board is probably obvious. The Society's work is expanding into many new areas and there should be representation from a number of these on the Board from time to time. A larger Board also will help in the work of the Board's subcommittees by permitting somewhat larger subgroups and avoiding duplication of membership.

To effectuate the proposed new requirement for 18 Directors, it is expected the 1959 Nominating Committee will present nominees for eight Directors instead of the previous five, designating by selection or by lot two who would serve respectively for one and two-year terms. After this period, new Directors would have a normal three-year term.

A careful scrutiny of the Society's financial operations by the Long-Range Planning Committee and especially by the Finance Committee,. during which the importance and value of the Society's work to *industry* was emphasized, resulted in the recommendation that the annual dues for Company Members be increased from $50 to $75 and for Sustaining Members from $150 to $200 effective in 1959. The present privileges of these two classes will continue, including that of designating different technically qualified individuals to serve on diverse committees to which the company may be elected and, in the case of the Company Membership, that of receiving publications on the same basis as the individual.

Sustaining Members will continue to receive on request a copy of every book issued by the Society and also receive special consideration in connection with the Book of ASTM Standards and the ASTM Bulletin. This class of membership was established in the 1930's to enable the larger organizations and associations which benefit inherently more from the activities of ASTM than do individuals and smaller companies to help keep ASTM finances on a sound basis and permit the steady expansion of ASTM services. Currently there are almost 400 Sustaining Members.

As with most operations, the cost of operating the Society is on the increase; and since printing and publications costs have increased drastically in the past five years, it was felt the need for these nominally increased dues would be understood. With the constantly increasing number of publications, membership is also increasing in value.

No changes are currently contemplated in the dues for Individual, Associate, or Student Members.

The following revision in the By-laws will be required:

Article II. Officers and Their Election

SECTION 1. Amend Section 1 by the deletion of the word in parentheses and the addition of the italicized word:

> "The officers of the Society shall be a President, two Vice-Presidents, (fifteen) *eighteen* Directors, and an Executive Secretary."

SEC. 2. Amend the last sentence of Section 6 by the deletion of the word in parentheses and the addition of the italicized word:

> "The Directors shall hold office for three years and shall be so classified that the terms of office of (five) *six* Directors shall expire each year."

Article VIII. Dues

Amend the second sentence of Section 1 by the deletion of the figures in parentheses and the addition of the italicized figures:

> "The annual dues, payable in advance, shall be as follows: For Individual Members, $18; for Company Members, ($50) *$75*; for Sustaining Members, ($150) *$200*; for Associate Members, $10; for Student Members, $2."

The above revisions are subject to action at the Annual Meeting and letter ballot approval of the Society. If approved at the Annual Meeting, they will be included as items on the letter ballot to be sent to each member after the Annual Meeting.

Membership

TO insure the Society's growth not only numerically but also along technical lines, companies and individuals must be acquainted with our work and asked to participate. Thus we are obviously concerned with membership growth. This has not been spectacular but is constantly increasing in all classes.

On May 1, 1958, the membership of the Society totaled 9359, compared with a total of 8836 one year ago. Gains and losses in various classes of membership are shown in the following table:

Class of Membership	Membership		Losses				Additions		Total		
	May 1, 1957	May 1, 1958	Resignations	Dropped	Death	Transfer	Transfer	Election	Loss	Gain	Increases
Honorary	25	29	...	...	...	...	2	2	...	4	4
Perpetuity and Life	13	13	...	...	...	...	...	...	...	...	...
Sustaining	382	417	2	...	...	5	39	3	7	42	35
Company, Firm, etc.	1967	2041	63	9	...	44	16	174	116	190	74
Individual, etc.	6263	6603	329	90	46	13	16	802	478	818	340
Associate	186	256	15	7	...	11	...	103	33	103	70
Total	8836	9359	409	106	46	73	73	1084	634	1157	523
Student	806	1031	87	405	...	29	...	746	521	746	225

It is interesting to compare our present membership of 9359 with the figures for ten and twenty years ago; in 1948 our total membership was 6438, and in 1938 it was 4156.

In 1957 there were almost as many new members as in the previous high year, 1956—1002 compared to 1014. The average in several previous years was from 602 to 775. Our net gain in 1957 was 523.

The number of company and sustaining members continues to grow.

Associate and Student Memberships:

The class of Associate Membership, which extends to 30 years of age and replaced the former junior membership with its 27-year age limit, seems to have met with favor, considering the substantial growth in this class, as noted in the table.

Because of the influence of student membership prize awards, which recognize outstanding students in courses related to the Society's work, there has been a very encouraging growth in this class. The Society itself and many of the districts, in addition to individuals and companies, are sponsoring student membership awards.

Sustaining Members:

There have been 42 new sustaining members in the period from May, 1957, to May, 1958, and seven resignations or transfer to company memberships. The new sustaining members are:

American Machine and Foundry Co.
American Metal Climax, Inc.
American Potash and Chemical Co., Whittier Research Laboratory
American Window Glass Co.
Archer-Daniels-Midland Co.
Atlas Powder Co. Library
Bell Aircraft Corp.
C. F. Braun and Co.
Columbia Steel and Shafting Co.
Commercial Solvents Corp.
Darling Valve and Manufacturing Co.
Driver-Harris Co.
Electric Hose and Rubber Co.
Fibreboard Paper Products Corp.
General Cable Corp.
General Radio Co.
Handy & Harman
Holyoke Wire and Cable Corp.
I-T-E Circuit Breaker Co.
Kennecott Copper Corp.
Kimberly-Clark Corp.
Kuhlman Electric Co.
The Martin Co.
National Petro-Chemicals Corp.
Neptune Meter Co.
Nichols Wire and Aluminum Corp.
Nopco Chemical Co.
Northwestern Steel and Wire Co.
Orenda Engines, Ltd.
Charles Pfizer and Co., Inc.
Polymer Corp., Ltd.
Portland Gas and Coke Co.
The Richardson Co.
The Ruberoid Co.
Simplex Wire and Cable Co.
Solvay Process Division, Allied Chemical Corp.
Tele-Dynamics, Inc.
Titan Metal Manufacturing Co.
Triangle Conduit and Cable, Inc.
Underwriters' Laboratories, Inc.
Union Wire Rope Corp.
U. S. Rubber Co., Research Center

50-Year Members:

There are eight individuals and twelve organizations as noted below that this year have been connected with the Society continuously for 50 years.

H. Austill
Roland P. Davis
Henry A. Gardner
Prévost Hubbard
Albert J. Loepsinger
Frank M. Masters
Charles E. Paul
H. H. Scofield

Brown University, Division of Engineering
Columbia Steel and Shafting Co.
Crane Co.
General Electric Co.
Abbot A. Hanks, Inc.
International Paper Co.
Laboratorio Central de Ensayo de Materiales de Construccion, Escuela de Caminos (Spain)
Pecora Paint Co.
Purdue University Library
Joseph T. Ryerson and Son, Inc.
Union Drawn Steel Div., Republic Steel Corp.
United States Pipe and Foundry Co.

These members will be recognized at the Annual Meeting.

40-Year Members:

There are 35 members who this year have completed 40 years of membership, bringing the total number of certificates issued to the "Forty-Year Club" to 418.

The members who will receive the 40-Year Certificates at this Annual Meeting are as follows:

W. & T. Avery, Ltd.
Bohn Aluminum and Brass Corp.
J. G. Bragg
The Bristol Brass Corp.
Robert H. Brown
Clay Products Assn.
Columbia University Civil Engineering Research Laboratories
Daniel Cushing
Watson Davis
Delco Appliance Div. of General Motors Corp.
The Dow Chemical Co.
Malcolm Duncan
General Electric Co., Ltd., Witton Works
The Glidden Co., Paint Division
Granite City Steel Co.
The Imperial Inst.
Iowa Malleable Iron Co.
The M. W. Kellogg Co.
William B. Kouwenhoven
Lebanon Steel Foundry
Lock Joint Pipe Co.
Louisville & Nashville Railroad Co.
James T. MacKenzie
Magnus Metal Div., National Lead Co.
Missouri-Kansas-Texas Railroad Co.
National Association of Purchasing Agents
R. L. Sanford
Seaboard Air Line Railroad Co.
Sinclair Refining Co.
The Symington-Gould Corp.
The Texas & Pacific Railway Co.
Union Oil Co. of California
James S. Vanick
H. H. Wermine
Raymond W. Woodward

Deaths:

The Society has lost the following 46 members through death (figures in parentheses are dates of membership):

Adams, Comfort A. (1920)
Baker, W. E. Byron (1949)
Becker, William D. (1950)
Bennett, J. W. (1945)
Breeze, Lewis K. (1953)
Broughton, William Waite (1955)
Brown, Warren G. (1943)
Cook, Leon D., Jr. (1947)
Craig, R. W. (1952)
Davis, E. T. (1952)
Du Comb, William C. (1902)
Dunning, R. E. (1948)
Evans, Beverley A. (1955)
Flickinger, L. C. (1948)
Forbes, Duncan P. (1942)
Fraser, W. R. (1937)
Gordon, B. T. (1954)
Hayes, T. D. (1945)
Jameson, Arthur H. (1917)
Jasper, T. McLean (1924)
Kahn, Noah A. (1935)
Kennedy, William A. (1941)
Kepler, F. M. (1948)
Lutts, Carlton G. (1918))
MacLeod, Albert (1951)
Mattimore, H. S. (1914
McCreath, Lesley (1908)
Merica, Paul D. (1915)
Naudet, P. T. (1954)
Olcott, Floyd B. (1934)
Olmstead, Frank R. (1954)
Piatt, William M. (1940)
Preston, G. William (1938)
Riedel, John Charles (1921)
Schlick, W. J. (1920)
Settle, J. E. (1953)
Small, Ben John (1956)
Smith, G. D. (1952)
Smull, L. C. (1951)
Steger, A. K. (1954)
Thatcher, Raymond L. (1951)
Thulman, Robert K. (1954)
Walford, J. B. (1951)
Walter, L. W. (1903)
Welch, L. E. (1942)
Wiltsee, William P. (1920)

In addition to the above, the following representatives of company members of the Society passed away:

R. L. Albrook, Washington State Institute of Industrial Research
G. H. Anderson, New England Power Service Co.
F. G. Campbell, Elgin, Joliet and Eastern Railway Co.
A. R. Carr, Wayne State University
E. L. Cook, Seaboard Air Line Railroad Co.
B. S. Court, Hayes Steel Products, Ltd.
J. H. Crawford, Merrimack College
M. C. Dailey, Kaiser Gypsum Co., Inc.
J. W. Deaderick, American Lava Corp.
F. X. Gilg, The Babcock & Wilcox Co.
C. A. Gutenkunst, Jr., Milwaukee Malleable and Grey Iron Works
G. B. Hatch, The Texas Company

E. D. Johnson, City of Muskegon, Engineering Dept.
H. L. Kennedy, Dewey & Almy Chemical Co.
W. R. Kuenzel, Deering, Milliken and Co., Inc.
R. C. Palmer, Newport Industries, Inc.
Harold Rosenbloom, Thompson and Co.
J. J. White, Erie Forge and Steel Corp.
Howard R. Youngkrantz, Apex Smelting Co.

A number of these men have been affiliated with the Society for many years and rendered outstanding services, particularly through work on the technical committees. The various issues of the ASTM BULLETIN have included appropriate notices and appreciation of their work.

Honors and Awards

MANY men have given outstanding service to the Society and its fields of work. Each year ASTM honors men through election to national office and honorary membership, giving awards of merit, and recognizing outstanding technical contributions.

Descriptions of the various awards as well as the personnel of the lecture and award committees are given in the ASTM Year Book.

Recipients of honors and awards to be given at the 1958 Annual Meeting are indicated below:

Honorary Membership:

H. J. Ball (semi-retired), Professor of Textile Engineering, Lowell Technological Inst.
T. S. Fuller (retired), long-time Engineer in Charge of the Works Laboratory, General Electric Co.
J. L. McCloud (retired), long-time Director of Chemical Engineering and Chemical and Metallurgical Research, Ford Motor Co.
J. G. Morrow, Metallurgical Engineer, The Steel Co. of Canada, Ltd.

Award of Merit:

O. P. Beckwith
T. I. Coe
R. H. Heilman
C. P. Larrabee
F. V. Lenel
J. J. Moran
J. B. Rather, Jr.
A. H. Scott
B. F. Scribner
M. A. Swayze
L. J. Trostel
V. P. Weaver

Recognition of Technical Papers:

Charles B. Dudley Medal (research).—O. D. Sherby, T. A. Trozera, J. E. Dorn for their paper "The Effects of Creep Stress History at High Temperatures on the Creep of Aluminum Alloys."

Richard L. Templin Award (testing).—H. F. Robertson, D. Gross, J. Loftus for their paper "A Method for Measuring Surface Flammability of Materials Using a Radiant Energy Source."

Sam Tour Award (corrosion).—W. G. Renshaw, R. A. Lula for their paper "The Corrosion Properties of Chromium-Nickel-Manganese Austenitic Stainless Steel."

Sanford E. Thompson Award (concrete, sponsored by Committee C-9).—T. S. Chang, C. E. Kesler for their paper "Correlation of Sonic Properties of Concrete with Creep and Relaxation."

C. A. Hogentogler Award (soils, sponsored by Committee D-18).—D. M. Burmister for his paper "Application of Environmental Testing of Soils."

Recognition of Outstanding Contributions:

Max Hecht Award (industrial water, sponsored by Committee D-19).—F. R. Owens, President and General Manager, Cyrus William Rice and Co., Inc., Pittsburgh, Pa.

Committee D-12 Award (soaps and other detergents).—F. W. Smither, Chemist, National Bureau of Standards, Washington, D. C. (retired), former chairman of Committee D-12.

Harold DeWitt Smith Memorial Medal (textiles).—S. J. Kennedy, Research Director, Textile, Clothing, and Footwear Division, Quartermaster Research and Development Center, Natick, Mass.

ASTM Lectures:

The *Edgar Marburg Lecture* on "Man and Raw Materials" will be presented on Wednesday afternoon, June 25, at the Hotel Statler, Boston, Mass., by E. W. Pehrson, Chief, Division of Foreign Activities, U. S. Bureau of Mines, Washington, D. C. The *H. W. Gillett Memorial Lecture* on "High Temperature Metals—Their Role in the Technological Future" will be presented on Tuesday afternoon, June 24, also at the Hotel Statler, by Clyde Williams, President, Clyde Williams and Co., Columbus, Ohio (formerly President, Battelle Memorial Inst.).

Finances

THE Finance Committee recognizes the importance of sound finances in underwriting much of the constantly expanding work. The Society's financial condition has for years been relatively healthy. This is attributable to the excellent support received from industry, and also to the value of ASTM service—publications, standards, research data.

Report for 1957:

Full details of the fiscal operations of the Society for 1957 are included in the report of the auditors appearing in Appendix I.[1]

Operating Receipts:

Our expectations that 1957 would be a good year income-wise were more than realized. The estimated receipts of $943,000 actually developed to be $1,020,700. Of this amount, receipts from dues and entrance fees were $283,400 or 27.7 per cent; receipts from sales of publications were $624,000 or 61.2 per cent; and receipts from miscellaneous sources consisting principally of advertising, income from investments, exhibits and registration fees totaled $113,300 or 11.1 per cent.

It will be noted that receipts from membership fees are again, for the fourth consecutive year, under 30 per cent of the total.

Operating Disbursements:

Total disbursements were $1,028,200 which included $75,000 for the 1958 Book of Standards reserve and $83,000 to pay for the 1957 *Proceedings* and Book of Standards Supplements.

A total of $431,000 was spent for publications, which compares with $342,323 for the previous year. Expenditures for salaries, which included extra summer help and considerable overtime, reached $360,200. This figure represents 35.0 per cent of total disbursements. Cost of employee benefits, including the major medical policy and covering new participants in the retirement plan, totaled $47,700.

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[1] See p. 65.

Balance Sheet:

On November 30 our total assets in General Funds were $1,017,100 compared to $819,600 a year ago. In all funds—General, Building, Medal, etc.—total assets were $1,663,500 compared with $1,473,516 a year ago. It should be pointed out that in the Balance Sheet no account has been taken of the assets of the Society in the form of publications in stock. This is consistent with many years' practice in this respect and, of course, results in a more conservative statement. An inventory of the principal technical publications as of May 1, 1958, is summarized below:

Proceedings (All back copies from 1955)	2 716
1955 Book of ASTM Standards	
Part 1	511
Part 2	4
Part 3	140
Part 4	384
Part 5	675
Part 6	26
Part 7	698
1956 Supplement to Book of ASTM Standards	
Part 1	2 593
Part 2	1 451
Part 3	1 545
Part 4	1 245
Part 5	1 333
Part 6	1 114
Part 7	1 186
1957 Supplement to Book of ASTM Standards	
Part 1	3 158
Part 2	1 881
Part 3	6 600
Part 4	1 356
Part 5	1 472
Part 6	971
Part 7	1 166
ASTM Methods of Chemical Analysis	4 069
Special Compilations of Standards	19 366
Selected Standards for Use in College Curricula	2 943
Symposiums, STP's and Special Reprints	207 026
Reprints of Standards (Approx.)	250 000
Radiographic Standards for Steel Castings	15
Radiographic Standards for Aluminum & Magnesium Castings	17
Radiography of Steel Welds	43

Not included in inventory:

(1) Viscosity Temperature Charts
(2) Hardenability Charts and Petroleum Charts
(3) Knock Test Manual and Supplement
(4) Marburg Lectures
(5) Cotton Yarn Boards

Building Fund:

Assets in the Building Fund, which include the current Race Street Headquarters, the Cherry Street property, and the balance of the lot immediately adjoining this, total $308,400.

The loan from ASTM General Funds which enabled the procurement of the Cherry Street property now amounts to $38,025 as of November 30, 1958. It is being amortized in the amount of $8700 yearly and should be wiped out accordingly in about 5 years.

Investments:

In the operation of the Investment Fund, the Society had continuing advice from our investment counsel, Loomis, Sayles & Co. This Fund was set up in 1956 by transferring to it assets from the various operating funds such as General, Building, Research, etc., with these funds owning *pro rata* a share in the Investment Fund. The operating funds proportionately benefit from income and growth of the Fund. In January, 1958, the operating funds transferred further assets in the amount of $15,500.

During the year a number of changes in investments were made, some bonds and stocks being sold and a number of additional securities purchased. These are detailed in the Auditors' Report which is appended.[1]

As of November 30, the book value or cost of the securities in the fund totaled about $787,500; market value was $875,400. Total income was $38,600.

In recent months purchases have been made approximately in the ratio of holdings which prevailed in the fund in late

1956, namely, about 46 per cent bonds, preferreds and cash; 54 per cent common stocks.

The Society's holdings of *mutual funds* are included in the above totals of the Investment Fund. As of November 30 mutual holdings had a market value of about $187,500.

1958 Budget:

From the accompanying summary of the 1958 budget, it will be seen that a deficit is anticipated for the current year. This is a relatively normal expectation because our operations really are on a three-year basis, influenced by the triennial Book of Standards, and the income from the 1955 Book will fall off sharply during this year whereas it was at a peak in 1956, thus creating the very favorable balance in that year. Such funds as are necessary to make up the difference between income and disbursements will be drawn from the Society's surplus.

BUDGET SUMMARY—1958.

		Per cent
Estimated Receipts:		
Dues and Entrance Fees	$287 000	27.0
Publications	660 500	62.0
Miscellaneous	117 500	11.0
TOTAL CURRENT RECEIPTS	$1 065 000	100.0
FROM SURPLUS	45 000	
	$1 110 000	
Estimated Disbursements:		
Publications	$451 000	40.6
Salaries	395 000	35.6
General Office Expenses	101 000	9.1
Technical Committee Expenses	12 000	1.1
Meetings	30 500	2.7
Headquarters Occupancy Expenses	41 000	3.7
Retirement Insurance Expense	48 500	4.4
Miscellaneous Expenses	31 000	2.8
TOTAL OPERATING DISBURSEMENTS	$1 110 000	100.0

Cooperative Activities

COOPERATION is a keyword in advancing ASTM research and standards. It avoids duplication of effort; it pools various resources; it spreads the load of work. ASTM experience is invaluable in bringing together consumer and producer, in establishing close contacts with other societies and associations, and in aiding liaison between industry and government.

Other Societies and Associations:

There are upwards of 250 leading societies and associations which are members of ASTM, many participating actively through technically qualified representatives in committee work. Actually the representatives of many of these groups serve very capably as technical committee officers. Our cooperative activities run the gamut of joint sponsorship of technical committees, cooperation on individual standards or groups of standards, joint sponsorship of extensive technical symposiums, and numerous activities where large numbers of societies act together in the interest of a great number of engineers and scientists.

Federal and State Governments:

Close contacts are maintained with those responsible for standards work or coordination thereof, in both the Federal and State Governments, the most notable instances being with the Standards branches of the Department of Defense, and the General Services Administration, the latter responsible for Federal Specifications work. Since its inception 60 years ago, the Society has endeavored to coordinate standards work of industry and government. Several ASTM technical committees have standing subcommittees whose responsibility is to review on request proposed government specifications and tests to the mutual advantage of both groups.

There are groups of state highway officials, Government, and state purchasing officials, and others, all of whose work is important in coordinating buying requirements. ASTM is in close touch with these groups.

Some very pertinent remarks were made by former President R. A. Schatzel in his June, 1957, Presidential Address entitled "Our Expanding Technology and ASTM." Referring to ASTM and the Federal Government he said:

> "Our system of standardization is inherently tied to our system of free enterprise. It is a completely voluntary system, in which our Government may have representatives on committees, often acting as chairmen, and as at present, in our Society, members of government agencies serve on our Board of Directors. As a most important customer, the Government's representatives have great weight, but the Government does not try to dominate our work nor that of any other standardizing group on which it has representation.
>
> "It is also a fact that no single agency acts for the Government.
>
> "Although as far as we know, there is no single agency established on a national level to coordinate the needs of the Federal Government with those of industry as far as standards are concerned, ASTM has for many years, in fact, from its very inception, cooperated closely with those Governmental agencies that develop standards."

American Standards Association:

As one of five founders of the ASA, the Society has consistently supported the work of this federation of standards-writing bodies. Recently ASTM renewed its third member on the Council, thus adding additional financial support. However, the most significant relationship is the large number of ASTM standards which have been submitted to ASA for its approval. These now total upwards of 570, constituting about one third of the total approved by ASA.

Inter-American Conference on Standards:

Two of the Society's national officers, Director A. A. Bates and Executive Secretary R. J. Painter, were members of the 7-man official delegation representing the United States at the Inter-American Conference on Standards sponsored by the Organization of American States held in Rio de Janeiro in September, 1957. The general purposes of this conference were to expedite the formation of national standardizing groups in the various Latin-American countries and to establish ways and means of coordinating standards issued by the respective countries. The United States Committee will continue as a group advisory to the officials directing Latin-American activities. The recommendations are of considerable significance to many segments of industry. They were summarized in the December, 1957, ASTM BULLETIN.

Miscellaneous—International Critical Tables; President's Conference; Research Support:

Specific reference can appropriately be made to a few special developments during the year. First, ASTM is officially represented on an Advisory Board on Critical Tables, established by the National Academy of Science. Much of ASTM work involves the development of scientific and engineering data, and a few of our committee activities particularly will be of service in developing information that will in part bring up to date and supplement the so-called International Critical Tables issued many years ago. M. V. Otis, Tennessee Eastman Co., Head of the Subcommittee on Standard Data of Committee E-13 on Absorption Spectroscopy, is representing the Society.

ASTM accepted President Eisenhower's invitation to be represented at the Conference on Technical and Distribution Research for the Benefit of Small Business. ASTM President R. T. Kropf attended this two-day conference, the results of which are just now being published. Many so-called small business companies can be helped by participating in the work of their respective trade associations and by having their technical people or representatives maintain contacts with professional and technical societies.

Where materials are of concern, as in so many cases, the work of the Society is of interest.

While the Board of Directors has generally refrained from approaching Congressional committees on legislative matters, occasionally there are instances where in the interest of engineering and science it is felt a contact is appropriate. One area involves the expansion of the work of the National Bureau of Standards. So much of this work is vital in providing basic data and a great deal of it invaluable in promoting a better knowledge of materials that communications were addressed to the Chairman of the Senate and House Appropriations Committee urging that, if at all feasible, additional support be given to the Bureau.

Respectfully submitted on behalf of the Board of Directors,

RICHARD T. KROPF,
President.

R. J. PAINTER,
Executive Secretary.

June, 1958

APPENDIX I

REPORT OF THE AUDITORS FOR THE FISCAL YEAR DECEMBER 1, 1956 TO NOVEMBER 27, 1957

Philadelphia, December 18, 1957

MR. R. J. PAINTER, *Executive Secretary*
AMERICAN SOCIETY FOR TESTING MATERIALS
Philadelphia, Pa.

Dear Sir:

We have examined the books and accounts of the American Society for Testing Materials for the period December 1, 1956, to November 27, 1957. We did not make a detailed audit of all transactions, but made tests to the extent we considered appropriate in determining the accuracy of the accounts.

Cash on deposit in checking and savings accounts was verified by direct correspondence with the various depositories and reconciled with the cash records. We verified by direct correspondence the publication accounts receivable (200 accounts), and the replies received disclosed only insignificant differences. The distribution of income and the determination of participations in the principal of the investment fund were made as of November 15, 1957; therefore, all schedules and the market value of the securities of the investment fund are reflected as of November 15, 1957. A minor amount of income of $12.70 was not distributed; this amount will be included in subsequent distribution of income. The securities held by the investment fund as scheduled on pp. 77–78 hereof, and certificates of deposit were examined by us on December 18, 1957. Income from investments for the period under review was verified by us and found to be properly recorded on the books of the Society.

During the current fiscal period the Society adopted a new contributory pension plan effective May 1, 1957. Members of the previous retirement plan have agreed to become a member of this plan and have assigned their rights under the old plan to the trustees of the new plan. Employees' contributions are equal to 2.5 per cent of annual compensation subject to Social Security (presently $4200), and 3.75 per cent of annual compensation in excess thereof. The remaining cost necessary to fund the benefits under the plan will be contributed by the Society and it is estimated that such cost to the Society will approximate five times the employees' contribution. The contribution made by the Society in the current period amounted to $35,000. The plan was approved by the Treasury Department as a qualified trust under Section 401(*a*) of the Internal Revenue Code by letter dated November 5, 1957. The plan may be amended or discontinued by the Society.

We have prepared and submit herewith balance sheet as of November 27, 1957, and statement of cash receipts and disbursements and operations for the period December 1, 1956 to November 27, 1957, and other supporting schedules which, in our opinion, present fairly the financial position of the Society at November 27, 1957, and the results of its operations for the period ended that date.

Respectfully submitted,
JOHN HEINS & CO.

BALANCE SHEET AS OF NOVEMBER 27, 1957

(Including Special and Designated Funds)

ASSETS

General Funds:				
Current:				
Cash			$279 307.37	
Investment Fund Participation (at cost)			597 007.33	
Accounts Receivable			62 814.44	
Loans Receivable (Building Fund)			38 025.00	
Advance to Forest Products Laboratory			500.00	
Deferred Expense			2 475.80	
Total Current Assets—General Funds			980 129.94	
Furniture and Fixtures (depreciated book value)			36 965.25	
Total Assets—General Funds				$1 017 095.19
Building Fund:				
Cash			11 377.71	
Investment Fund Participation (at cost)			25 551.73	
Land, Cost			68 459.42	
Buildings and Construction Costs		$276 600.28		
Less, Accumulated Depreciation		73 600.00	203 000.28	
Total Assets—Building Funds				308 389.14
Other Special and Designated Funds:				
Cash:				
ASTM Research Fund		11 866.36		
Dudley Medal and Marburg Lecture Fund	$505.99			
Richard L. Templin Award Fund	214.74			
Sam Tour Award Fund	345.72			
F. E. Richart Award Fund	45.37			
Total Medal and Lecture Funds		1 111.82		
Investment Fund		12.70		
Committee Funds		82 492.13	95 483.01	
Investment Fund Participation (at cost):				
ASTM Research Fund		73 096.75		
Dudley Medal and Marburg Lecture Fund		6 968.07		
Richard L. Templin Award Fund		2 229.79		
Sam Tour Award Fund		1 735.44		
F. E. Richart Award Fund		1 051.78		
Committee Funds		108 077.31	193 159.14	
Other Investments (at cost:)				
ASTM Research Fund		10 074.63		
Committee Funds		39 325.00	49 399.63	
Total Assets—Special and Designated Funds				338 041.78
Total Assets				$1 663 526.11

LIABILITIES

General Funds:			
Current:			
Advance Collections		$17 221.56	
Accounts Payable:			
Committee C-1		116.63	
Joint Committee on X-Ray Diffraction		190 472.76	
Joint Symposium, Nuclear Meeting		1 476.77	
Pennsylvania Sales Tax		77.61	
Provision for:			
1957 Proceedings		36 000.00	
1957 Supplements to Book of Standards		47 000.00	
Total Current Liabilities—General Funds		292 365.33	
Life Membership Fund	$4 017.94		
Publication Fund	16 841.80		
Book of Standards Reserve	161 030.02		
Executive Retirement Reserve	15 973.60		
Retirement Fund Reserve	37 334.68		
Reserve for Depreciation of Investments	20 000.00		
Reserve for Additional Cost of Replacement of Headquarters Building	35 000.00		
Special Annual Meeting Entertainment Account	4 661.64		
	294 859.68		
Surplus	429 870.18	724 729.86	
Total Liabilities and Surplus—General Funds			$1 017 095.19
Building Fund:			
Contributions from Members		183 953.05	
Contributions from ASTM General Fund		82 064.57	
Notes Payable—General Fund		38 025.00	
Accumulated Income, Net		4 346.52	
Total Building Fund			308 389.14
Other Special and Designated Funds:			
ASTM Research Fund:			
Principal	84 926.75		
Income	10 110.99	95 037.74	
Dudley Medal and Marburg Lecture Fund:			
Principal	6 968.07		
Income	505.99	7 474.06	
Richard L. Templin Award Fund:			
Principal	2 229.79		
Income	214.74	2 444.53	
Sam Tour Award Fund:			
Principal	1 735.44		
Income	345.72	2 081.16	
F. E. Richart Award Fund:			
Principal	1 051.78		
Income	45.37	1 097.15	
Investment Fund—Undistributed Income		12.70	
Committee Funds, Unexpended Balance		229 894.44	
Total Special and Designated Funds			338 041.78
Total Liabilities and Funds			$1 663 526.11

In Addition to the General Funds of the Society the Treasurer Had on Hand the Following Funds as of November 27, 1957

COMMITTEE FUNDS

Committee A-1 on Steel	$2 465.71
Committee A-5 on Corrosion of Iron and Steel	478.34
Committee B-3 on Corrosion of Non-Ferrous Metals and Alloys	2 983.14
Committee B-5 Weaver Award Fund	17.40
Committee B-6 on Die-Cast Metals and Alloys	3 237.50
Committee B-8 on Electrodeposited Metallic Coatings	14.12
Committee C-1 on Cementitious, Ceramic, Concrete, and Masonry—General Funds	1 884.15
Committee C-1 Cement Reference Laboratory	876.26
Committee C-9 Sanford E. Thompson Medal Fund	1 118.25
Committee C-9 on Concrete and Concrete Aggregates	225.31
Committee C-15 on Manufactured Masonry Units	534.20
Committee C-18 on Natural Building Stone	437.60
Committee D-1 on Paint, Varnish, Lacquer, and Related Products	618.27
Committee D-2 on Petroleum Products and Lubricants	1 962.68
Committee D-2 ASTM—IBM	1 082.15
Committee D-2 (DCC) Reference Fuel Account	35 263.63
Committee D-2 (DCC) National Exchange Group	(584.42)[a]
Committee D-4 on Road and Paving Materials	1 011.10
Committee D-5 on Coal and Coke	839.13
Committee D-7 on Wood	50.82
Committee D-7 Wood Pole Research Fund	17 135.71
Committee D-9 on Electrical Insulating Materials	2 818.87
Committee D-10 on Shipping Containers	11.95
Committee D-11 on Rubber—Special	592.46
Committee D-11 on Rubber and Rubber-Like Materials	367.59
Committee D-12 on Soaps and Detergents	1 090.73
Committee D-13 on Textile Materials	1 171.63
Committee D-14 on Adhesives	57.79
Committee D-15 on Engine Antifreezes	544.03
Committee D-18 on Soil Testing Procedures—Hogentogler Award Fund	16.60
Committee D-19 Max Hecht Award Fund—Industrial Water	147.41
Committee D-20 on Plastics	1 169.76
Committee E-2 on Emission Spectroscopy	131.08
Committee E-3 on Chemical Analysis of Metals	204.14
Committee E-4 on Metallography	66.55
Committee E-9 on Fatigue	285.85
Committee E-14 on Mass Spectrometry	2 267.70
ASA Sectional Committee on Specifications for Cast Iron Pipe	1 583.57
Chicago District	3 675.25
Cleveland District	265.89
Detroit District	2 479.10
Ohio Valley District	4.61
Philadelphia District	831.07
Philadelphia District Special (Annual Meeting Account)	2 443.19
Southern California District	3 241.79
Northern California District	2 007.49
Reserve—Future West Coast Meetings	7 172.46
Pittsburgh District	259.70
Washington District	201.17
Western New York – Ontario District	1 935.60
Advisory Committee on Corrosion	48 089.94
Administrative Committee on End-Use Products	291.73
Joint Committee on X-Ray Diffraction, Chemical Analysis	57 346.64
Joint Committee on X-Ray Diffraction, Research Fellowship	15 367.75
Joint Committee on X-Ray Diffraction, Indexing Patterns—IBM Calculator	102.30
	229 894.44

Accounted for as Follows:

Cash in Bank and on Hand		$82 492.13	
Investments (at cost)—(market value $38 137.50):			
$20 000 U. S. Treasury 2½s due 8/15/63	$19 925.00		
20 000 U. S. Treasury 2½s due 11/15/61	19 400.00	39 325.00	
Investment Fund Participation		108 077.31	$229 894.44

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[a] ( ) Indicate red figures.

DUDLEY MEDAL AND MARBURG LECTURE FUND

Principal Account

	Total	*Invested*
Balance, December 1, 1956	$6 943.33	$6 943.33
Receipts:		
Transfer from Income—Stock Dividends	19.80	19.80
Transfer from Income—Gain on Sale of Investments	7.07	7.07
Loss on Sale of Investments	(2.13)[a]	(2.13)[a]
	$6 968.07	6 968.07

Income Account

Balance, December 1, 1956	705.13	
Receipts:		
Interest, Dividends, and Gain on Investments	322.28	$1 027.41
Disbursements:		
Transfer to Principal Account	26.87	
Award	400.00	
Medals	79.10	
Certificates	15.45	521.42
Balance, November 27, 1957		$505.99

RICHARD L. TEMPLIN AWARD FUND

Principal Account

			Uninvested Cash
Balance, December 1, 1956, as reported	$2 221.87	$2 221.87	
Receipts:			
Transfer from Income—Stock Dividends	6.34	6.34	
Transfer from Income—Profit on Sale of Investments	2.26	2.26	
Loss on Sale of Investments	(0.68)[a]	(0.68)[a]	
Balance, November 27, 1957	$2 229.79	2 229.79	

Income Account

Balance, December 1, 1956, as reported	172.99	
Receipts:		
Interest, Dividends, and Gain on Investments	103.13	$276.12
Disbursements:		
Transfer to Principal Account	8.60	
Awards	50.00	
Certificates	2.78	61.38
Balance, November 27, 1957		$214.74

SAM TOUR AWARD FUND

Principal Account

Balance, December 1, 1956, as reported	$1 729.28	$1 729.28
Receipts:		
Transfer from Income—Stock Dividends	4.93	4.93
Transfer from Income—Gain on Sale of Investments	1.76	1.76
Loss on Sale of Investments	(0.53)[a]	(0.53)[a]
Balance, November 27, 1957	$1 735.44	1 735.44

Income Account

Balance, December 1, 1956	309.92	
Receipts:		
Interest, Dividends, and Gain on Investments	80.27	$390.19
Disbursements:		
Transfer to Principal Account	6.69	
Awards	35.00	
Certificates	2.78	44.47
Balance, November 27, 1957		$345.72

ASTM RESEARCH FUND

Principal Account

	Total	*Invested*	*Uninvested Cash*
Balance, December 1, 1956, as reported	$81 851.28	$74 595.91	$7 255.37
Receipts:			
Gift of 16 Sh American Telephone & Telegraph Co.	2 816.00	2 816.00	
Transfer from Income—Stock Dividends	207.62	207.62	
Transfer from Income—Gain on Sale of Investments	74.19	74.19	
Loss on Sale of Investments	(22.34)[a]	(22.34)[a]	
Additional Funds Transferred to Investment Fund		5 500.00	(5 500.00)[a]
Balance, November 27, 1957	84 926.75	83 171.38	1 755.37
Investment Fund Participation	73 096.75		
618 Sh General Motors Co. (Common) (market value $22 634.25)	7 258.63		
16 Sh American Telephone & Telegraph Co. (market value $2 648.00)	2 816.00	83 171.38	

Income Account

Balance, December 1, 1956, as reported	8 357.07	
Receipts:		
Interest, Dividends, and Gain on Sale of Investments	3 380.83	11 737.90
Disbursements:		
Transfer to Principal—Stock Dividends and Gain on Investments	281.81	
American Electro Platers Society—Research Fund	500.00	
Corrosion Research Council of the Engineering Foundation	300.00	
Committee D-15 on Engine Antifreezes	500.00	
Printing	45.10	1 626.91
Balance, November 27, 1957		$10 110.99

F. E. RICHART AWARD

Principal Account

Balance, December 1, 1956	$1 048.05	$1 048.05	
Receipts:			
Transfer from Income—Stock Dividends	2.98	2.98	
Transfer from Income—Gain on Sale of Investments	1.07	1.07	
Loss on Sale of Investments	(0.32)[a]	(0.32)[a]	
Balance, November 27, 1957	$1 051.78	1 051.78	

Income Account

Balance, December 1, 1956		91.23	
Receipts:			
Interest, Dividends, and Gain on Investments		48.64	
Contribution from ASTM		50.00	$189.87
Disbursements:			
Transfer to Principal Account		4.05	
Awards		125.00	
Certificates		15.45	144.50
Balance, November 27, 1957			$45.37

GENERAL FUNDS: RECEIPTS AND DISBURSEMENTS

FOR THE PERIOD DECEMBER 1, 1956 TO NOVEMBER 27, 1957

Cash Balance, December 1, 1956, as reported			$126 447.80
RECEIPTS			
Operating Receipts (Budgeted):			
Dues and Entrance Fees:			
Current Dues	$272 681.15		
Life Membership	175.00		
Past Dues	1 544.57		
Advance Dues	2 732.80		
Entrance Fees	6 255.00	$283 388.52	
Sales of Publications:			
Book of Standards (Members, Additional Parts)	61 409.15		
1952 Book of Standards	482.37		
1955 Book of Standards	136 552.49		
1953 and 1954 Supplements to Book of Standards	121.05		
1956 Supplement to Book of Standards	85 059.32		
1957 Supplement to Book of Standards	670.00		
Methods of Chemical Analysis of Metals	16 715.10		
Compilation of Standards	109 145.65		
Separate Standards	41 630.51		
Selected Standards for Students	7 282.15		
Proceedings	11 101.09		
Bulletin Subscriptions (Nonmembers)	4 714.74		
Special and Misellaneous Publications	149 052.85	623 936.47	
Miscellaneous:			
Advertising—BULLETIN	50 457.60		
Advertising—Index	3 853.23		
Advertising—1958 Index	15.00		
Interest and Dividends	30 492.16		
Registration and Other Fees—Meetings	16 404.02		
ASTM Exhibit at Annual Meeting	2 950.00		
Furniture and Fixtures	113.71		
Staff Service	8 236.74		
Miscellaneous	832.49	113 354.95	
Total Operating Receipts (Budgeted)		1 020 679.94	
Nonoperating Receipts (Not Budgeted):			
Excess Remittances	4 796.17		
Committee C-1 for Technical Assistant	2 599.98		
Retirement Fund Reserve	1 226.43		
Sale of X-Ray Diffraction Cards	131 273.92		
British and Metric I P Tables	437.84		
Institute of Petroleum Publications	63.84		
From Building Fund on Account of Loan	8 700.00		
From Investment Fund Repayment of Advance	36 000.00		
Annual Meeting Luncheon	1 756.00		
Annual Meeting Buffet Supper	316.25		
ASTM—Joint Symposium Meeting	4 297.50		
Gain on Sale of Securities	605.90		
Investments	25 000.00		
Miscellaneous	119.44	217 193.27	
Total Receipts			1 237 873.21
Total Receipts for Fiscal Year 1957 and Cash Balance December 1, 1956			1 364 321.01

DISBURSEMENTS

Operating Disbursements (Budgeted):

Publications:			
Year Book	$13 875.12		
1955 Book of Standards	5 563.88		
1958 Book of Standards	3 147.20		
1956 Supplement to Book of Standards	4 836.40		
1957 Supplement to Book of Standards	1 963.29		
Compilations of Standards	37 541.72		
Separate Standards	12 921.47		
Methods of Chemical Analyses of Metals	10 993.61		
Proceedings—Repurchase of Old Numbers	11.64		
1955 Proceedings	86.81		
1956 Proceedings	93.32		
1957 Proceedings	8 606.77		
Preprints	11 668.63		
ASTM BULLETIN	56 859.06		
Circulars to Members	2 685.41		
Index to Papers	3 277.91		
Index to Standards	9 324.92		
Special and Miscellaneous and West Coast Publications	88 718.30	$272 175.46	
Salaries	360 171.19		
General Office Expenses	93 079.30		
Expenses—Technical and District Committees	10 105.62		
Expenses—Meetings	18 948.82		
Headquarters Occupancy Expense (Includes $9,800 Depreciation on Buildings)	41 088.72		
American Standards Association	1 500.00		
Traveling Expense, Administrative and Special Committees	6 675.28		
Furniture and Fixtures	9 460.66		
Dues, Contributions, Miscellaneous	1 734.71		
Group Insurance	1 941.16		
Employees' Retirement Fund	37 995.89		
Social Security	6 106.76		
Blue Cross	1 484.49		
Major Medical	2 144.10		
Legal, Actuarial, and Related Services	5 550.00	597 986.70	
Total Operating Disbursements (Budgeted)		870 162.16	
Total Receipts and Cash Balance December 1, 1956			$1 364 321.01

Nonoperating Disbursements (Not budgeted):

Refund of Excess Remittances	4 792.27		
Technical Assistant Committee C-1	2 629.98		
Costs and Refunds, X-Ray Diffraction Cards	20 991.81		
Transfer to Joint Committee on X-Ray Diffraction	50 000.00		
Institute of Petroleum Publications	4 466.01		
Annual Meeting Buffet Supper	408.55		
Annual Meeting Luncheon	973.50		
Executive Retirement Reserve	1 200.00		
Gillett Memorial Fund	75.00		
Insurance, Corrosion Test Site	110.00		
Employee Retirement Reserve	290.64		
ASTM—Joint Symposium	2 820.73		
Investments	84 825.06		
Accrued Interest	233.82		
Advance to Investment Fund	36 000.00		
Awards Luncheon	1 286.15		
Special Annual Meeting Account	925.00		
Standards Conference, Rio de Janeiro	2 438.55		
Meeting, Mexico City	37.25		
Miscellaneous	347.16	214 851.48	
Total Disbursements			1 085 013.64
Cash Balance, November 27, 1957			$279 307.37

ASTM BUILDING FUND

DETAILS OF ASTM BUILDING FUND CASH RECEIPTS AND DISBURSEMENTS FOR THE PERIOD DECEMBER 1, 1956 TO NOVEMBER 27, 1957

Cash Balance, December 1, 1956		$16 646.75

Receipts

Contributions from Members	$2 898.63	
Interest, Dividends, and Profit on Investments	1 181.80	
From ASTM General Fund:		
On Account of Depreciation on Building, Elevator, and Air Conditioning Installation	9 800.00	
On Account of Amortization of Note	8 700.00	
Salvage	15.00	22 595.43
		39 242.18

Disbursements

Expenses, Cherry Street Property:		
Real Estate Taxes, 1957	2 711.09	
Construction	9 850.10	
Expenses, Race Street Property:		
Construction	1 964.77	
Loading Zone Permit	40.00	
Reduction of Loan from ASTM General Fund	8 700.00	
Transferred to Investment Fund	4 598.51	27 864.47
Cash Balance, November 27, 1957		$11 377.71

DETAILS OF ASSETS AND LIABILITIES OF BUILDING FUND AS OF NOVEMBER 27, 1957

Assets

Cash in Girard Trust Corn Exchange Bank, Philadelphia, Pa.				$11 377.71
Investment Fund Participation (at cost)				25 551.73
Property 1916–1918 Race St., Philadelphia, Pa:				
Cost:				
Land			$30 000.00	
Building		$26 503.35		
Construction (Net of Salvage) to November 30, 1956		135 331.06		
Additions 1957:				
Partitions		483.77		
Cooling Tower		1 481.00		
		163 799.18		
Accumulated Depreciation (Provided from General Funds)		60 083.05	103 716.13	
			133 716.13	
Property 1921–1927 Cherry St. and 1922–1928 Quarry St., Philadelphia, Pa:				
Cost:				
Land		30 800.00		
Buildings	$38 880.10			
Construction to November 30, 1956	63 180.43			
Additions, 1957:				
Lights and Decorating	2 229.11			
Replacing Stairways	1 591.30			
Alterations McGonigal Building	6 029.69			
	111 910.63			
Accumulated Depreciation (Provided from General Funds)	13 516.95	98 393.68	129 193.68	
Property 1919 Cherry St., Philadelphia, Pa:				
Cost, Land		7 659.42		
Improvements		890.47	8 549.89	271 459.70
Total Assets				$308 389.14

Liabilities and Funds

Contributions from Members	$183 953.05	
Contributions from ASTM Funds	82 064.57	
Accumulated Net Income	4 346.52	
Loan Payable to ASTM General Funds	38 025.00	$308 389.14

ASTM INVESTMENT FUND PARTICIPATIONS

AS OF NOVEMBER 15, 1957

Cash	$28 182.35	
Investments (at cost) (Market value $875 396.85)	787 548.55	
Total Assets		$815 730.90
Participation by Funds	815 718.20	
Undistributed Income	12.70	$815 730.90

	November 30, 1956 Participation	*Additional Funds Invested*	*Income Reinvested*	*Net Profit, Sale of Securities*	*Total Participation November 15, 1957 (At Cost)*
Participations by Funds:					
General Fund	$537 388.15	$57 500.00	$1 695.72	$423.46	$597 007.33
Committee Funds	100 193.67	7 500.00	306.98	76.66	108 077.31
Research Fund	67 337.28	5 500.00	207.62	51.85	73 096.75
Building Fund	20 961.02	4 500.00	72.58	18.13	25 551.73
Medal and Lecture Fund	6 943.33		19.80	4.94	6 968.07
Templin Award Fund	2 221.87		6.34	1.58	2 229.79
Sam Tour Fund	1 729.28		4.93	1.23	1 735.44
Richart Fund	1 048.05		2.98	.75	1 051.78
	$737 822.65	$75 000.00	$2 316.95	$578.60	$815 718.20

ASTM INVESTMENT FUND

DETAILS OF INVESTMENT FUND CASH RECEIPTS AND DISBURSEMENTS FOR THE PERIOD DECEMBER 1, 1956 TO NOVEMBER 15, 1957

Cash Balance, December 1, 1956			$28 463.54
Receipts			
Interest and Dividends on Investment	$38 593.44		
Interest on Savings Accounts	1 068.83		
Interest on Certificates of Deposit	350.00		
Total Interest and Dividends		$40 012.27	
Proceeds from Sales of Investments	39 386.65		
Additional Funds Invested by Participants	75 000.00		
Reinvestment of Stock Dividends by Participants	2 316.95		
Loan from General Funds	36 000.00	152 703.60	
Total Receipts			192 715.87
			221 179.41
Disbursements			
Purchase of Securities		117 596.85	
Investment Counsel		2 500.00	
Repayment of Loan from General Funds		36 000.00	
Distribution of Interest and Dividends to Participants:			
General Fund	27 006.50		
Committee Funds	4 889.04		
Research Fund	3 306.64		
Building Fund	1 155.87		
Medal and Lecture Fund	315.21		
Templin Award Fund	100.87		
Sam Tour Fund	78.51		
Richart Fund	47.57	36 900.21	
Total Disbursements			192 997.06
Cash Balance, November 15, 1957			28 182.35
Balance Composed of:			
Demand Account		15 529.63	
Savings Account		2 652.72	
Certificates of Deposit		10 000.00	$28 182.35

NOTE: On November 22, 1957 $15,000 Consumers Power 4¾s, 1987 were purchased for $15,294.38 including accrued interest of $106.88 entered on books of the Society as a 1958 fiscal year transaction.

INVESTMENT FUND

AS OF NOVEMBER 15, 1957

Investments

Number of Shares or Par Value	*Bonds*	*Cost or Book Value*	*Market Value*
$3 000	Atlantic Refining Co. 4½ per cent Conv. Deb. 8/15/87	$3 000.00	$3 090.00
10 000	Commercial Credit Co. 4½ per cent 1977	9 806.00	8 500.00
15 000	Consolidated Edison Co. of N. Y., Inc. 1st and Ref. 3⅝ per cent Series L 5/1/86	15 206.55	12 975.00
10 000	Detroit Edison Co. 3¾ per cent Conv. Deb. 9/14/71	11 000.00	11 300.00
15 000	General Motors Corp. 3¼ per cent Deb. 1/1/79	14 925.00	13 350.00
1 000	General Telephone Corp. 4 per cent Conv. Deb. 5/1/71	1 001.25	950.00
10 000	General Telephone Corp. 4½ per cent Conv. Deb. 6/1/77	10 681.23	9 937.50
5 000	Pacific Gas & Electric Co. 1st and Ref. 3⅛s Series W 12/1/84	5 150.78	4 075.00
10 000	Radio Corp. of America 3½ per cent Conv. Deb. 12/1/80	11 075.00	8 600.00
20 000	Scott Paper Co. 3 per cent Conv. Deb. 3/1/71	20 377.80	18 400.00
5 000	Sinclair Oil Corp. 4⅜ per cent 1986	5 731.68	5 125.00
15 000	Socony Mobil Co. 2½ per cent Deb. 6/1/76	13 995.95	12 150.00
15 000	Southern Natural Gas Co. 1st Pipe Line S F 3⅛s 2/1/74	14 734.81	12 000.00
15 000	Superior Oil Co. 3¾ per cent Deb. 7/1/81	15 000.00	13 650.00
10 000	Tennessee Gas Transmission Co. 1st Mtg Pipe Line 4¾s 11/1/76	10 065.00	9 900.00
10 000	U. S. Federal Land Banks 3⅞ per cent 9/15/72	9 806.25	9 587.50
5 000	U. S. Twelve Federal Land Banks 3¾ per cent 7/15/57		
15 000	U. S. Twelve Federal Land Banks 2¾ per cent 5/1/58	14 850.00	14 906.25
10 000	U. S. Twelve Federal Land Banks 3⅞s 2/14/58	9 995.00	9 993.75
15 000	U. S. Twelve Federal Land Banks 4⅛s 2/15/72	15 000.00	14 775.00
2 000	U. S. Treasury 2½s 6/15/64–69	2 000.00	1 803.75
5 000	U. S. Treasury 2½s 3/15/65–70	5 000.00	4 493.75
15 000	U. S. Treasury 2½s 11/15/61	15 000.00	14 362.50
8 500	U. S. Treasury 2½s 6/15/67–72	8 632.83	7 575.63
15 000	U. S. Treasury 2½s 8/15/63	14 943.75	14 240.63
15 000	U. S. Treasury 2¾s 9/15/61	14 934.38	14 531.25
20 000	U. S. Treasury 4s 8/1/61	20 056.25	20 387.50
30 000	U. S. Savings Bonds Series G 2½s 6/1/61	30 000.00	29 100.00
5 000	U. S. Savings Bonds Series G 2½s 1/1/62	5 000.00	4 820.00
5 000	U. S. Savings Bonds Series G 2½s 2/1/62	5 000.00	4 820.00
	Total Bonds	331 969.51	309 400.01

Preferred Stocks

Shares			
30	Endicott Johnson Corp. 4 per cent Pfd	2 937.50	2 310.00
30	General Motors Corp. $5	3 755.80	3 165.00
30	Long Island Lighting Co. 4.35 per cent Series E Pfd	3 015.00	2 340.00
30	P. Lorillard Co. 7 per cent Pfd	4 380.00	3 660.00
30	Mississippi Power & Light Co. 4.36 per cent Cum Pfd	3.075.00	2 400.00
100	Philadelphia Electric Co. 4.40 per cent Cum Pfd	10 670.00	9 400.00
47	Standard Slag Co. 2nd Pfd	2 115.00	2 115.00
30	Westinghouse Electric Corp. 3.80 per cent Cum Pfd Series B	3 067.74	2 475.00
	Total Preferred Stocks	33 016.04	27 865.00

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[b] All market values of securities are as of November 15, 1957.

Common Stocks

Shares			
202	Allegheny Ludlum Steel Corp.	$8 111.87	$7 196.25
100	Aluminum Co. of America	2 061.45	6 650.00
100	American Can Co.	3 884.25	3 762.50
300	American Cyanamid Co.	6 238.91	11 887.50
100	American Radiator & Standard Sanitary Co.	1 564.32	1 137.50
166	American Stores Co.	7 638.83	9 960.00
56	American Telephone and Telegraph Co.	8 919.72	9 268.00
150	Atchison, Topeka & Santa Fe Railroad Co.	2 961.06	2 681.25
160	Bethelehem Steel Corp.	2 095.99	6 260.00
230	Carpenter Steel Co.	6 104.75	11 040.00
136	Chase Manhattan Bank	3 930.06	6 120.00
200	Cleveland Electric Illuminating Co.	8 271.00	6 650.00
150	Climax Molybdenum Co.	8 879.80	8 700.00
100	Crown Zellerbach Corp.	2 914.79	4 462.50
75	Delaware Power and Light Co.	2 454.11	3 431.25
170	Dow Chemical Co.	3 997.75	9 137.50
50	E. I. duPont de Nemours & Co.	6 544.18	8 700.00
50	Florida Power & Light Co.	1 969.44	2 500.00
125	General Electric Co.	4 311.15	7 609.38
150	General Mills, Inc.	10 630.89	8 456.25
225	General Telephone Corp.	3 312.82	9 000.00
100	B. F. Goodrich Co.	5 140.42	5 875.00
100	Great Northern Paper Co.	10 045.00	4 612.50
135	Gulf Oil Corp.	3 476.68	15 153.75
238	Houston Lighting & Power Co., Texas	8 061.31	12 673.50
200	International Harvester Co.	6 406.67	6 100.00
200	Johns-Manville Corp.	8 063.78	7 400.00
103	Jones & Laughlin Steel Corp.	2 636.06	4 866.75
500	Lone Star Cement Corp.	4 941.44	14 437.50
50	Louisville & Nashville Railroad Co.	4 154.59	2 950.00
200	Montgomery Ward & Co.	5 798.04	6 250.00
103	Niagara Mohawk Power Corp.	3 479.57	2 922.63
200	Ohio Oil Corp.	2 623.35	5 975.00
60	Pacific Gas & Electric Co.	2 301.42	2 820.00
50	Parke, Davis & Co.	1 641.19	3 000.00
321	Philadelphia Electric Co.	8 599.20	11 475.75
200	Pittsburgh Consolidation Coal Co.	8 760.94	6 050.00
100	Republic Steel Corp.	2 152.93	4 525.00
200	Seaboard Oil Co.	11 956.88	11 500.00
154	Sears Roebuck Co.	3 168.32	3 907.75
100	Sherwin Williams Co.	10 515.69	11 800.00
138	Socony Mobil Oil Co.	3 103.03	6 503.25
150	Square D Co.	2 362.37	3 412.50
200	Standard Oil of Indiana	11 969.64	7 500.00
273	Standard Oil of New Jersey	8 526.48	13 513.50
80	Texas Co.	2 184.35	4 940.00
60	Union Carbide Corp.	4 235.24	5 632.50
240	Westinghouse Electric Co.	15 644.26	14 400.00
200	Wisconsin Electric Power Co.	6 788.63	5 800.00
	Total Common Stocks	275 534.62	350 606.76

Mutual Funds

Shares			
849	Broad Street Investing Corp.	11 337.87	16 759.26
4 350.449	Commonwealth Investment Co.	31 793.03	34 933.50
5 376	Eaton Howard Balanced Fund	83 433.04	105 477.12
2 176	Fundamental Investors, Inc.	20 464.44	30 355.20
	Total Mutual Funds	147 028.38	187 525.08

Summary

Bonds	331 969.51	309 400.01
Preferred Stocks	33 016.04	27 865.00
Common Stocks	275 534.62	350 606.76
Mutual Funds	147 028.38	187 525.08
	$787 548.55	$875 396.85

Note: Income includes proceeds from sales of rights.

APPENDIX II

REPORT OF ADMINISTRATIVE COMMITTEE ON STANDARDS

The Administrative Committee on Standards is interested in all aspects of the Society's standardization work. The several functions are discussed under the subdivision headings of this report.

Actions by Standards Committee on Committee Recommendations:

The review of recommendations from technical committees is, of course, one of the primary functions of the Standards Committee, since in its establishment it was intended to provide a means of having recommendations acted upon in the interval between Annual Meetings. Many of the annual reports refer to recommendations that have been submitted to the Standards Committee during the year. The actions taken this year by the Standards Committee are summarized below:

New Tentatives	26
Revised Tentatives	74
Tentative Revisions of Standards	6
Standards Revised and Reverted to Tentative	15
Emergency Alternates Withdrawn	2

A number of these recommendations were acted upon at a meeting held on September 13, 1957; the remainder were handled by correspondence.

Actions by the Society at 1957 Annual Meeting:

As a matter of record, the actions taken by the Society at the 1957 Annual Meeting with respect to standards and tentatives are tabulated below:

Existing Tentatives Adopted as Standard	64
Standards Revised	88
New Tentatives	90
Standards Revised and Reverted to Tentative	18
Tentative Revisions of Standards	15
Tentatives Revised	140
Standards and Tentatives Withdrawn	11

Standard Certification Clauses:

Many ASTM specifications refer to certification of product in one form or another. The Standards Committee has been cognizant for some time of the variations that exist in the manner in which this certification is handled. It has been felt there should be some uniformity in this regard to make sure all of the essential aspects are provided for. After very careful consideration, the Standards Committee has developed suggested wordings as set forth below covering the use of certification. The Standards Committee recommends that they be brought to the attention of all technical committees for their information and guidance.

Inspection by Lot.—Technical committees should define the lot to facilitate inspection and testing of materials specified in standards under their jurisdiction.

The Waiving of Inspection by Agreement.—(*a*) When agreed upon in writing by the purchaser and the supplier, a certification shall be made the basis of acceptance of the material. This shall consist of a copy of the manufacturer's test report or a statement by the supplier, accompanied by a copy of the test results, that the material has been sampled, tested, and inspected in accordance with the provisions of the specification specified. Each certification so furnished shall be signed by an authorized agent of the supplier or manufacturer.

(*b*) When original identity cannot be established, certification can only be based on the sampling procedure provided by the applicable specification.

EXPANSION OF STANDARDIZATION ACTIVITIES

An important function of the Standards Committee is to explore with the Board of Directors proposals dealing with new projects. Comments with respect to a number of these are set forth below.

Industrial Chemicals.—As indicated in previous reports, the subject of industrial chemicals is so broad that considerable study is required to delineate the field more specifically. The Board of Directors has appointed a small committee to serve as a continuing study and advisory committee in the area of industrial chemicals. There is now being recommended the establishment of a new Committee E-15 on Analysis and Testing of Industrial Chemicals with the thought that the immediate approach in this broad field should be concentrated on methods of testing, particularly such methods as may have a rather general application.

Cleaning Solvents.—The report of a year ago mentioned considerations that were being given to requests that had been received that work be undertaken in the field of cleaning solvents, both from the point of view of cleaners for electric motors and also other types of cleaning such as those utilizing vapor degreasers. Although consideration was given to organizing a committee from the point of view of use, the conference referred to in that report recommended that a new committee be established covering Halogenated Organic Solvents. This recommendation has been approved by the Board of Directors, and the organization meeting of the committee, designated D-26, is being held during the 1958 Annual Meeting of the Society.

Flexible Barrier Materials.—In the report of a year ago, reference was made to a conference that was being called of representatives from the industry as well as from the various technical committees that would seem to have an interest in flexible barrier materials. At the time it was felt that a liaison committee to coordinate the activities of the several ASTM committees was needed. On the recommendation of the general conference, the Board of Directors authorized a new Committee on Flexible Barrier Materials. This committee, which has been given the designation of F-2, held its organization meeting on December 12, 1957.

Cermets.—The previous report referred to the establishment of a study committee to explore the need for any concerted work in the general field of cermets and to indicate how best such work might be organized if needed. This study committee has been organized and has held two meetings during the year. One of the primary considerations in the investigations of this committee is to arrive at some understanding as to what is normally considered as falling under the classification of cermets. Considerable additional exploratory work would seem to be needed before any specific recommendations can be agreed upon.

Gaskets and Packing Materials.—The Society has a long history with respect to its work on gasketing and packing materials. This has been concentrated in Committee D-11 on Rubber and Rubber-like Materials since many of the materials had a rubber base. Much of the work was carried on in cooperation with the Society of Automotive Engineers under a Joint SAE-ASTM Committee on Automotive Rubber. In view of the very broad interest in gaskets, much broader than automotive alone, and also the inclusion of types of materials other than organic-base materials covered in Committee D-11, a general conference was called to determine whether the present coverage by the Society is adequate. One possibility would be the establishment of a separate committee on gaskets and packing materials. By action of this conference a special study committee has been established so that specific recommendations can be formulated.

Abrasives.—Although the subject has often received consideration, at no time has there been any recommendation that a technical committee in the abrasives field be organized. Recently a specific request has been received from the Abrasive Grain Association for the preparation of an ASTM method covering density of abrasive grains. In the absence of a technical committee on abrasives, this has been referred to Committee E-1 on Methods of Testing.

RELATIONS WITH AMERICAN STANDARDS ASSOCIATION

Two methods under the procedure of the American Standards Association are used by ASTM in submitting recommendations to the ASA. One is a combination of the so-called existing standards procedure and the proprietary sponsorship procedure. On the initial reference of an existing ASTM standard to the ASA, it is submitted under the existing standards procedure with a supporting statement giving a history of the development of the standard and indicating the degree of its acceptance. If the standard is approved as American Standard, the ASTM is granted proprietary sponsorship insofar as any future revisions are concerned, and such revisions are brought to the attention of the ASA currently for approval under this proprietary sponsorship procedure.

The second method consists of having standards reviewed in a sectional committee made up of representatives of a number of interested organizations, usually with the ASTM as sponsor or cosponsor of the sectional committee.

Standards Submitted Under the Existing Standards Procedure:

Forty-six standards relating to wood were submitted to the American Standards Association for approval as American Standard under the existing standards procedure.

Revised Standards Submitted Under the Proprietary Procedure:

In view of the Society having been designated as proprietary sponsor for the revisions of ASTM standards that had been approved as American Standard under the existing standards procedure, it has submitted to the American Standards Association during the year revisions of 11 standards relating to steel, 2 relating to corrosion of iron and steel, 2 relating to copper, 3 relating to clay pipe, 2 relating to gypsum, 1 relating to refractories, 7 relating to manufactured masonry units, 2 relating to paint pigments, 1 relating to rubber products, 2 relating to detergents, and 1 relating to thermometers.

Standards Submitted Under the Sectional Committee Procedure:

During the year, the following sectional committees, for which the Society is sponsor or cosponsor, took action to present recommendations to the ASA as follows:

Sectional Committee B36 on Dimensions and Materials of Wrought-Iron and Wrought-Steel Pipe and Tubing.—This committee has recommended the approval of revisions of 18 American Standards.

Sectional Committee C7 on Bare Electrical Conductors.—This committee has recommended the approval as American Standard of 4 ASTM standards, and the approval of revisions of 17 American Standards.

Sectional Committee C59 on Electrical Insulating Materials.—This committee has recommended the approval of revisions of 5 American Standards.

Sectional Committee Z11 on Petroleum Products and Lubricants.—This committee has recommended the approval as American Standard of 4 ASTM standards, and the approval of revisions of 12 American Standards.

INTERNATIONAL RELATIONS

The international discussions, particularly under the International Organization for Standardization (ISO), are assuming ever greater significance. A number of the European countries apparently wish to have the standardization discussions handled internationally, and much of the work is supported here rather than in the development of individual national standards. In certain areas these international discussions are having a definite impact on the standardization in this country, and a number of ASTM committees are following these international discussions quite closely with the thought that the ASTM standards and the international recommendations should be in harmony. A list of the various ISO projects in which ASTM is interested and which continues to grow year by year is given below.

Aromatic Hydrocarbons.—ASTM Committee D-16 on Industrial Aromatic Hydrocarbons and Related Materials is giving consideration to American participation in the work of ISO/TC 78 on Aromatic Hydrocarbons for which Great Britain holds the secretariat.

Asbestos-Cement Products.—ASTM Committee C-17 on Asbestos-Cement Products has indicated that it favors American participation in the work of ISO/TC 77 on the basis of an observer. Switzerland holds the secretariat for this ISO committee.

Cast Iron.—Great Britain holds the secretariat for ISO/TC 25 on Cast Iron. The Advisory Committee of ASTM Committee A-3 on Cast Iron has been set up to handle American participation.

Chemistry.—Italy holds the secretariat for ISO/TC 47 on Chemistry. Several ASTM committees have indicated an interest in this work, and draft proposals which have been received have been passed on to these committees.

Copper and Copper Alloys.—The United States holds the secretariat for ISO/TC 26 on Copper and Copper Alloys. The Advisory Committee of ASTM Committee B-5 on Copper and Copper Alloys has been set up to handle the work of the secretariat.

Fiber Building Board.—A proposal had been received for the organization of a new ISO committee to deal with fiber building board. This received consideration in Committee D-7 on Wood, and based upon these considerations the United States through the American Standards Association voted negatively on the organization of this ISO committee. It received sufficient support, however, from other countries so that this committee has now been organized. The sentiment in Committee D-7 was that if the committee should be organized, the United States should participate in the work.

Fire Tests for Building Materials and Structures.—An inquiry has been received on participation on the part of the United States in an ISO committee to deal with fire tests of building materials and structures. ASTM Committee E-5 on Fire Tests of Materials and Construction has recommended against United

States participation on the basis that such participation could not be justified in view of the expense involved.

Hydraulic Binders.—Sectional Committee A1 on Hydraulic Cements has recommended participation by the United States in ISO/TC 74 on Hydraulic Binders. The sectional committee also has recommended that the group in this country to handle this participation be ASTM Committee C-1 on Cement.

Iron and Steel.—The United States has the status of an observer in the work of ISO/TC 17 on Iron and Steel for which Great Britain holds the secretariat. A number of draft proposals have been received which have been brought to the attention of Committee A-1 on Steel, while others dealing with methods of testing have been submitted to the appropriate subcommittees of Committee E-1 on Methods of Testing.

Laboratory Glassware.—Great Britain holds the secretariat for ISO/TC 48 on Laboratory Glassware. America is participating in this work through a special group on which ASTM is represented through Committee E-1 on Methods of Testing.

Light Metals and Alloys.—The Advisory Committee of ASTM Committee B-7 on Light Metals and Alloys has been set up to handle American participation in the work of ISO/TC 79 on Light Metals and Alloys. France holds the secretariat for this ISO committee.

Mica.—Subcommittee IX on Mica of ASTM Committee D-9 on Electrical Insulating Materials has been set up to handle the American contacts with ISO/TC 56 on Mica for which India holds the secretariat.

Paper.—ASTM Committee D-6 on Paper and Paper Products is giving consideration to American participation in the work of ISO/TC 6 on Paper. France holds the secretariat for this ISO committee.

Petroleum Products.—The United States holds the secretariat for ISO/TC 28 on Petroleum Products, the technical work of the secretariat being under the purview of Sectional Committee Z11 on Petroleum Products and Lubricants, for which ASTM is sponsor.

Plastics.—America holds the secretariat for ISO/TC 61 on Plastics. ASTM Committee D-20 on Plastics is handling the work of the secretariat and has organized for the purpose a special committee consisting of representatives of the various groups interested.

Raw Materials for Paints, Varnishes, and Similar Products.—ASTM Committee D-1 on Paint, Varnish, Lacquer, and Related Products is handling American contacts with ISO/TC 35 on Raw Materials for Paints, Varnishes, and Similar Products, for which the Netherlands holds the secretariat.

Refractories.—ASTM Committee C-8 on Refractories is handling American contacts with ISO/TC 33 on Refractories for which Great Britain holds the secretariat.

Rubber.—The secretariat for ISO/TC 45 on Rubber is held by Great Britain. The American group to handle participation in this ISO committee has been set up under ASTM Committee D-11 on Rubber and Rubber-like Materials.

Shellac.—Subcommittee XIII on Shellac of ASTM Committee D-1 on Paint, Varnish, Lacquer, and Related Products has been set up as the American committee in charge of contacts with ISO/TC 50 on Lac for which India holds the secretariat.

Solid Mineral Fuels.—ASTM Committee D-5 on Coal and Coke is handling the contacts of the United States with ISO/TC 27 on Solid Mineral Fuels for which Great Britain holds the secretariat.

Statistical Treatment of Series of Observations.—The Netherlands holds the secretariat for ISO/TC 69 on Statistical

Treatment of Series of Observations. The United States is participating in this work through a special group on which ASTM is represented.

Textiles.—Great Britain holds the general secretariat for ISO/TC 38 on Textiles, but the United States holds the secretariat for several subcommittees. American participation in this work is being handled by a special committee organized under the sponsorship of ASTM and the American Association of Textile Chemists and Colorists with representation from the various groups interested. This special committee has been given the designation of L23.

Timber.—ASTM Committee D-7 on Wood has indicated that it favors American participation in the work of ISO/TC 55 on Timber for which Russia holds the secretariat.

Viscosity.—The United States holds the secretariat for ISO/TC 66 on Viscosity. The Subcommittee on Rheological Properties of ASTM Committee E-1 on Methods of Testing has been set up to handle the work of the secretariat.

Zinc.—ASTM Committee B-2 on Non-Ferrous Metals and Alloys is in favor of United States participation in the work of ISO/TC 18 on Zinc.

Respectfully submitted on behalf of the committee,

J. H. FOOTE,
Chairman.

R. E. HESS,
Secretary.

APPENDIX III

REPORT OF ADMINISTRATIVE COMMITTEE ON PAPERS AND PUBLICATIONS

The work before the Committee on Papers and Publications continues to increase. In the 1957 report mention was made of the 200 papers presented at the 1956 West Coast meeting which constituted a particularly heavy load over and above the usual publications in a regular annual meeting year. But quite apart from this special meeting, the regular publications by and large are continuing to increase. For example, there are approximately 150 papers on the program for the 1958 Annual Meeting as compared with approximately 100 papers on the program for the 1957 Annual Meeting. In addition, there is an ever-increasing number of papers and articles submitted for publication in the ASTM BULLETIN, partly resulting from meetings sponsored by the local districts, but more particularly papers submitted specifically for BULLETIN publication without any request for presentation.

The review of material for publication constitutes an ever-increasing problem, not only because of the greater amount of material but also because of the necessity of having a more critical review of the material for publication. This critical review is based not only on the need for conserving the Society's funds by insisting on condensation and the restriction of published material to that which has definite merit, but also has in mind the conservation of the reader's time in locating material which will be of direct interest and benefit to him. Recognizing the increased burden on the individual members of the Papers Committee, the committee looks to outside reviewers to an ever-increasing extent and has sought the assistance of past members of the committee in handling some of the committee's responsibilities. For example, the Papers Committee is represented on each of the special symposium committees that are appointed to handle prospective programs at meetings, and in some instances this representation is handled by former members of the Papers Committee.

The extent of the Society's publications is indicated in the record of publications issued during the year.

1958 Annual Meeting:

As indicated above, the program for the 1958 Annual Meeting is an extremely heavy one. In all, some 43 sessions are contemplated; of these 18 will be devoted to special symposiums as follows:

Symposium on Basic Mechanisms of Fatigue,
Symposium on Some Approaches to Durability in Structures,
Symposium on Solvent Extraction in the Analysis of Metals,
Symposium on Bulk Sampling,
Symposium on Radiation Effects on Materials (Vol. 3),
Symposium on Effect of Water on Bituminous Paving Mixtures,
Symposium on Materials Research Frontiers,
Symposium on Particle Size Measurement,
Symposium on Paper and Paper Products,
Symposium on Applications of Soil Testing in Highway Design and Construction, and
Symposium on Radioactivity in Industrial Water and Industrial Waste Water.

In addition the several industrial luncheons may result in publication.

Book of ASTM Standards:

The consideration with respect to issuing the 1958 Book of ASTM Standards referred to in the 1957 report culminated in the decision that the 1958 book be issued in ten parts. These are as follows:

Part 1—Ferrous Metals (Specifications);
Part 2—Non-Ferrous Metals (Specifications), Electronic Materials;
Part 3—Methods of Testing Metals (Except Chemical Analysis);
Part 4—Cement, Concrete, Mortars, Road Materials, Waterproofing, Soils;
Part 5—Masonry Products, Ceramics, Thermal Insulation, Acoustical Materials, Sandwich and Building Constructions, Fire Tests;
Part 6—Wood, Paper, Adhesives, Shipping Containers, Cellulose, Leather;
Part 7—Petroleum Products, Lubricants, Tank Measurements, Engine Tests;
Part 8—Paint, Naval Stores, Aromatic Hydrocarbons, Coal and Coke, Gaseous Fuels, Engine Antifreezes;
Part 9—Plastics, Electrical Insulation, Rubber, Carbon Black; and
Part 10—Textiles, Soap, Water, Atmospheric Analysis, Wax Polishes.

It is expected that these ten parts should be adequate for at least three issues of the Book of Standards, with all volumes being a convenient size.

Year Book:

The publication of the Year Book continues to be a problem in view of its very rapid increase in size. As mentioned previously, some sections formerly included in the Year Book were eliminated, but of these the geographic distribution has been reinserted which of itself adds considerably to the size. Various suggestions are being explored to see if the material in the Year Book can be published on some different basis.

RECORD OF PUBLICATIONS ISSUED DURING THE YEAR

Regular Publications:

1957 Proceedings, 1440 pp., 7500 copies.
1957 Marburg Lecture, 40 pp., 3000 copies.
1957 Gillett Lecture, 36 pp., 3000 copies.
1957 Supplement to Book of Standards:
    Part 1, 528 pp., 14,500 copies.
    Part 2, 391 pp., 11,000 copies.
    Part 3, 436 pp., 9400 copies.
    Part 4, 230 pp., 7500 copies.
    Part 5, 359 pp., 7650 copies.
    Part 6, 380 pp., 7800 copies.
    Part 7, 320 pp., 7500 copies.
1957 Year Book, 756 pp., 7400 copies.
1957 Index to Standards, 251 pp., 28,000 copies.
ASTM BULLETIN, 8 issues (May 1957 to April 1958), total number of pages 840, average number of copies 14,937 (269 pages of technical papers).

A statement of the volume of the principal publications for the past four years is given below:

	1954–1955, pages	1955–1956, pages	1956–1957, pages	1957–1958, pages
Proceedings	1425	1128	1528	1440
Marburg Lecture*	....	54	51	40
Gillett Lecture*	....	....	32	36
Book of ASTM Standards	....	11 752	....	....
Supplements to Book of Standards	2746	....	2335	2644
ASTM Methods of Chemical Analysis of Metals	....	....	640	....
Index to ASTM Standards	306	216	240	251
Year Book	672	620	640	756
Special Technical Publications	2101	3018	3441	4061
Other Special Publications	46	964	878	208
ASTM BULLETIN	792	808	836	840
Totals	8088	18 560	10 538	10 276

* As of 1956 to be issued separately.

Special Compilations of Standards Published from April 15, 1957 to April 15, 1958:

Specifications for Steel Piping Materials (A-1), 455 pp., 3000 copies. (Published April 24, 1957.)

ASTM Standards on Cast Iron (A-3), 170 pp., 1200 copies.

ASTM Standards on Metallic Electrical Conductors (B-1), 344 pp., 900 copies.

ASTM Standards on Copper and Copper Alloys (B-5), 688 pp., 1600 copies. Additional reprints struck off, 1000 copies.

ASTM Standards on Light Metals and Alloys (B-7), 330 pp., 800 copies.

Manual of ASTM Standards on Refractory Materials (C-8), 420 pp., 4600 copies.

ASTM Standards on Gypsum Products, Plaster Aggregates and Related Standards (C-11), 148 pp., 2000 copies.

ASTM Standards on Glass and Glass Products (C-14), 146 pp., 1200 copies.

ASTM Standards on Petroleum Products and Lubricants (D-2), 1138 pp., 8750 copies.

ASTM Standards on Bituminous Materials for Highway Construction, Waterproofing and Roofing (D-4, D-8), 460 pp., 2000 copies. (Published June 28, 1957.)

ASTM Standards on Coal and Coke (D-5), 140 pp., 2000 copies.

ASTM Standards on Paper, Paper Products and Shipping Containers (D-6, D-10), 420 pp., 1600 copies.

ASTM Standards on Electrical Insulating Materials (D-9), 692 pp., 1200 copies.

ASTM Standards on Rubber and Rubber-Like Materials (D-11), 844 pp., 2400 copies. (Published June 28, 1957.)

ASTM Standards on Soaps and Other Detergents (D-12), 180 pp., 1200 copies.

ASTM Standards on Textile Materials (D-13), 870 pp., 2500 copies.

ASTM Standards on Adhesives (D-14), 252 pp., 1500 copies.

ASTM Standards on Engine Antifreezes (D-15), 56 pp., 1500 copies.

Manual on Industrial Water (D-19) (STP 148-C), 504 pp., 2250 copies. (Fourth printing.)

ASTM Methods for Emission Spectrochemical Analysis (E-2), 504 pp., 2500 copies.

ASTM Standards on Materials for Electron Tubes and Semiconductor Devices (F-1), 250 pp., 1500 copies.

Special Compilations in Prospect:

Specifications for Steel Piping Materials (A-1), 480 pp., 3000 copies.

ASTM Standards on Electrodeposited Metallic Coatings (B-8), 116 pp., 4000 copies.

ASTM Standards on Mineral Aggregates, Concrete and Nonbituminous Highway Materials (C-9, D-4), 384 pp., 2500 copies.

ASTM Standards on Paint, Varnish, Lacquer and Related Products (D-1), 1052 pp., 2000 copies.

ASTM Standards on Rubber and Rubber-Like Materials (D-11), 906 pp., 3000 copies.

ASTM Procedures for Testing Soils (D-18), 552 pp., 5000 copies.

Manual on Measurement and Sampling of Petroleum and Petroleum Products (D-2), 168 pp., 4000 copies.

Building Code Compilation, 972 pp., 3000 copies.

Special Publications Issued from April 15, 1597 to April 15, 1958:

ASTM Manual for Rating Motor Fuels by Motor and Research Methods, 88 pp., 2500 copies.

Bibliography of Fatigue References (STP 9-H), 64 pp., 500 copies—mimeographed.

ASTM Viscosity Tables for Kinematic Viscosity Conversions and Viscosity Index Calculations (STP 43-B), 66 pp., 5000 copies.

Index to the X-Ray Powder Data File (STP 48-G), 729 pp., 1000 copies.

Bibliography and Abstracts on Electrical Contacts (STP 56-K), 44 pp., 1500 copies.

1957 Supplement to the Metal Cleaning Bibliographical Abstracts (STP 90-D), 44 pp., 2000 copies.

Bibliographical Abstracts of Methods for Analysis of Synthetic Detergents (STP 150-B), 28 pp., 1500 copies.

Compilation of Chemical Composition and Rupture Strengths of Super Strength Alloys (STP 170-A), 5 pp., 1500 copies.

Symposium on Vane Shear Testing of Soils (STP 193), 76 pp., 2000 copies.

Symposium on Ion-Exchange and Chromatography in Analytical Chemistry (STP 195), 63 pp., 2000 copies.

* Papers on Metals (STP 196), 181 pp., 2500 copies.

* Symposium on Wood for Marine Use and Its Protection from Marine Organisms (STP 200), 56 pp., 2000 copies.

* Symposium on Structural Sandwich Construction (STP 201), 109 pp., 2500 copies.

* Symposium on Vapor Phase (STP 202), 74 pp., 2000 copies.

* Symposium on Fatigue with Specific Reference to Aircraft Structures (STP 203), 104 pp., 2000 copies.

* Symposium on Titanium (STP 204), 214 pp., 3000 copies.

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* 1956 West Coast Papers.

* Papers on Cement and Concrete (STP 205), 158 pp., 2500 copies.
* Papers on Soils (STP 206), 184 pp., 2000 copies.
* Symposium on Industrial Water and Industrial Waste Water (STP 207), 56 pp., 2500 copies.
* Symposium on Radiation Effects on Materials (Vol. 1) (STP 208), 196 pp., 3000 copies.
* Papers on Design and Tests of Building Constructions—Seismic and Shock Loading and Glued-Laminated Constructions (STP 209), 91 pp., 2500 copies.
* Symposium on Full Scale Tests on House Structures (STP 210), 64 pp., 2500 copies.
* Symposium on Steam Turbine Oils (STP 211), 112 pp., 2500 copies.
* Papers on Road and Paving Materials (STP 212), 82 pp., 2000 copies.
* Symposium on Nondestructive Testing (STP 213), 93 pp., 2500 copies.
* Symposium on Railroad Materials Including Lubricating Oils (STP 214), 175 pp., 2200 copies.
* Symposium on Radioisotopes (STP 215), 98 pp., 2500 copies.
Symposium on Large Fatigue Testing Machines and Their Results (STP 216), 157 pp., 2000 copies.
Symposium on Thermal Conductivity Measurements and Applications of Thermal Insulation (STP 217), 92 pp., 2500 copies.
Symposium on Insulating Oil (STP 218), 45 pp., 1500 copies.
Symposium on Determination of Dissolved Oxygen in Water (STP 219), 64 pp., 2000 copies.
Thermal Properties of Thirteen Metals (STP 227), 33 pp., 2000 copies.
Summary of Proceedings, 32 pp., 9200 copies.
Numerical List of Abstracted Ultraviolet Spectra-Indexed Wyandotte Cards, 88 pp., 500 copies.

Special Publications in Prospect:

Manual on Engine Test Methods—Aviation Supercharge, 256 pp., 1500 copies.
1950–1955 Spectrochemical Index, Part IV (STP 41-D), 230 pp., 2500 copies.
Bibliography and Abstracts on Electrical Contacts (STP 56-L), 48 pp., 1500 copies.
Symposium on Radiation Effects on Materials (Vol. 2) (STP 220), 143 pp., 2500 copies.
Symposium on Spectrochemical Analysis for Trace Elements (STP 221), 100 pp., 2000 copies.
Symposium on Determination of Gases in Metals (STP 222), 66 pp., 2000 copies.
Symposium on Nondestructive Tests in Nuclear Energy Field (STP 223), 401 pp., 3000 copies.
Symposium on Composition of Petroleum Oils (STP 224), 294 pp., 2500 copies.
Knocking Characteristics of Pure Hydrocarbons (STP 225), 88 pp., 3000 copies.
Report on Elevated Temperature Properties of Weld Deposits (STP 226), 228 pp., 3000 copies.
Report on Elevated Temperature Properties of Chromium Steel (STP 228), 100 pp., 3000 copies.
Symposium on Effect of Ozone on Rubber (STP 229), 80 pp., 2000 copies.
Symposium on Elevated Temperature Strain Gages (STP 230), 95 pp., 3000 copies.
Symposium on Brittle Failure of Rotor Forgings (STP 231), 64 pp., 2000 copies.
Conference Papers on Soils for Engineering Purposes (STP 232), 500 pp., 1200 copies.
† Symposium on Radiation Effects on Materials (Vol. 3) (STP 233), 180 pp., 2500 copies.
† Symposium on Particle Size Measurement (STP 234), 254 pp., 2500 copies.
† Symposium on Radioactivity in Industrial Water and Industrial Waste Water (STP 235), 68 pp., 2000 copies.
† Symposium on Some Approaches to Durability in Structures (STP 236), 70 pp., 2000 copies.
† Symposium on Basic Mechanisms of Fatigue (STP 237), 150 pp., 2500 copies.
† Symposium on Solvent Extraction in the Analysis of Metals (STP 238), 86 pp., 2000 copies.
† Symposium on Applications of Soil Testing in Highway Design and Construction (STP 239), 152 pp., 2000 copies.
† Symposium on Effect of Water on Bituminous Paving Mixtures (STP 240), 74 pp., 2000 copies.
† Symposium on Paper and Paper Products (STP 241), 92 pp., 2000 copies.
† Symposium on Bulk Sampling (STP 242).
† Symposium on Materials Research Frontiers (STP 243).
† Symposium on Stability of Distillate Fuel Oils (STP 244).
† Symposium on Advances in Electron Metallography (STP 245).

Respectfully submitted on behalf of the committee,

R. E. HESS,
Chairman.

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† 1958 Annual Meeting papers.

APPENDIX IV

REPORT OF ADMINISTRATIVE COMMITTEE ON DISTRICT ACTIVITIES

During the year every ASTM District held at least one meeting, making a total of 28. In addition, a special meeting was held in Albuquerque, N. Mex. Fifteen of these meetings were jointly sponsored with other technical organizations.

President R. T. Kropf delivered lectures on "Knowledge of Materials," "Textile Research," and "Future Needs for Technical Research" at ten district meetings. Everett P. Partridge, 1957 Marburg lecturer, spoke at Southwestern and Northern California District meetings on "Industrial Water." The 1957 Gillett lecturer, Alvin J. Herzig, addressed the Northern California, Southern California, and Detroit Districts on "Molybdenum and Its Promising Alloys."

Several districts have developed their technical programs in new and interesting ways. For example, the Chicago District met in conjunction with the Nuclear Congress; the Philadelphia District was host to a visiting delegation of the European Productivity Agency; and the New York District continued its program of having one meeting a year called a "Night at a Particular Company," this year being General Electric. Several districts, including the Southern California, Northern California, New York, New England, Washington, and Southwestern, have participated in Local Technical Societies Council affairs and Science Fairs. This has increased the awareness of other societies of the purposes of ASTM.

The Southeastern District was officially formed. Inaugural meetings were held in Columbus, Ga., and Shawmut, Ala. in November 1957, at which President R. T. Kropf and Executive Secretary R. J. Painter were present.

The proposed formation of a Central New York District Council, to comprise part of the former New York District, was approved by the Administrative Committee on District Activities and recommended to the Board of Directors for their approval. This step was taken in recognition of the need for formal Society organization in industrial areas geographically isolated from large centers of population.

Formation of a Rocky Mountain District, encompassing the states of New Mexico, Arizona, Colorado, Utah, Wyoming, and part of Western Texas, is under active study. A poll of members is being held following a very successful exploratory meeting in Albuquerque, N. Mex.

The Administrative Committee on District Activities recommended to the Board of Directors that revisions be made to the Charter and Manual for ASTM Districts to eliminate the requirement that annual reports be submitted by districts. This recommendation was subsequently approved. This step was taken in view of the fact that all pertinent information is currently supplied to Headquarters.

The Administrative Committee on District Activities recommended that each council appoint one member to handle local publicity. Of the 16 districts, 6 have appointed such public relations representatives, and publicity in local papers and technical journals has markedly increased.

At the request of several districts, the Administrative Committee on District Activities reviewed the provisions of the Charter for Districts relating to the size of District Councils. The committee has recommended changes to the Board of Directors to allow for larger councils where wider representation is desirable. However, it was stressed that the quality of District Council membership was of prime importance.

District Councils have been encouraged to form membership committees to stimulate Society membership. Only three councils have reported active committees, and further study in this area is contemplated by the Administrative Committee on District Activities.

The Students Awards Program reached an all-time high, with nine districts presenting over 200 individual awards. This year the Northern California, Western New York - Ontario, and New England Districts made awards for the first time. In addition, three other districts—Ohio, Washington, and St. Louis—have programs under study. The Cleveland District plans to renew them next year.

The St. Louis District was host to National Committee Week held in February 1958, and the New England District will be host to the Annual Meeting to be held in Boston. Two industry luncheons, for steel and chemicals, were held in St. Louis, together with a dinner featuring an address by William M. Holaday, Director of Guided Missiles, Defense Department.

The Administrative Committee on District Activities held a meeting during the 1957 Annual Meeting, at which J. H. Calbeck was appointed to fill a vacancy. The committee sponsored its annual breakfast meeting for District Council officers. Once again, it proved to be a valuable clearing house for information and practices used by the councils in their District affairs.

Respectfully submitted on behalf of the committee,

C. M. GAMBRILL,
Chairman.

ALBERT L. BATIK,
Secretary.

APPENDIX V

REPORT OF ADMINISTRATIVE COMMITTEE ON RESEARCH

Meetings of the Administrative Committee on Research were held on June 18, 1957, in Atlantic City, N. J., and on December 18, 1957, at ASTM Headquarters.

There were no changes in membership of this committee during 1957. A. T. McPherson and Frank T. Sisco were reappointed for three-year terms. Because of the wide interest that ASTM has in research in many fields, it is suggested that additional appointments be made to the present seven-man committee.

Financially, a modest gain of about $8000 was made in the research fund, bringing the total to about $85,000. Contributions totaling $1300 were made during the financial year to Committee D-15 on Engine Antifreezes, the Corrosion Research Council of the Engineering Foundation, and through Committee B-8 on Electrodeposited Coatings to finance cooperatively some research with the American Electroplaters Society Research Fund. At the request of Committee D-9 on Electrical Insulation, tentative authorization was also given to support a research project on testing of thermosetting laminates in cooperation with the National Electrical Manufacturers Assn. and Electronic Industries Assn.

Currently, the Administrative Committee on Research is giving considerable attention to the best way to put the Research Fund to work. The attention of technical committees is being called to the existence of the fund to assure that worthy projects which might be supported are called to the attention of the Administrative Committee.

Plans were made to sponsor a symposium on research at the 1958 Annual Meeting in Boston. This was in cooperation with the New England District Council Committee. The general subject of this symposium is "Materials Research Frontiers." It is believed that this symposium will be a real contribution to the excellence of the technical program as the wide range of materials to be discussed should have appeal to most of the Society membership.

A series of short articles by members of the Administration Committee were printed in the ASTM BULLETIN throughout the year. These "ACR Notes" generally cover items in which the committee is interested and which it is felt should receive the attention of the ASTM membership.

There has been only fair response from the technical committees in supporting or contributing to the listing of "Challenges in Materials Research" (formerly "Unsolved Problems"). Although calling attention to needed research has been a worthy objective, there is some doubt as to whether the good this project accomplishes outweighs the difficulties that have been encountered in promoting it.

One of the objectives of this committee is to make known the research activities of the Society. This is not easy since such activities cover a broad range of materials and it is often difficult to separate new knowledge gained by technical committee investigations from the general accumulation of information and experience in developing a test method or setting up a specification. The technical committees are being urged to review their research activities frequently and to publicize those items that are worthy of promotion as added knowledge of engineering materials. Currently, an effort is being made to survey thoroughly all research activities in progress or contemplated by the technical committees. An up-to-date "Review of ASTM Research Projects" is expected to be one result of this survey.

Periodically, the Administrative Committee reviews technical committee research projects that involve solicitation of funds. Sixteen projects of this nature were reviewed at the December 1957 meeting. Contact with the many researches of the National Research Council's Division of Engineering and Industrial Research is maintained through a Society representative. Likewise, close liaison is maintained with the activities of the Engineering Foundation.

Considerable attention was given at the last meeting of the Administrative Committee to questions posed by the Long Range Planning Committee of the Board of Directors. The whole subject of research funds and research encouragement is still actively being discussed, as well as means to make the Administrative Committee on Research more effective. Partly as a result of discussion on long range planning, a recommendation was made to the Board for the establishment of an Administrative Committee on Education. This would have as its chief function the establishment of a policy relating to all matters of the Society's contacts with universities.

Respectfully submitted on behalf of the committee,

BRUCE W. GONSER,
Chairman.

FRANK Y. SPEIGHT,
Secretary.

APPENDIX VI

REPORT OF ADMINISTRATIVE COMMITTEE ON SIMULATED SERVICE TESTING

Two meetings were held during the past year: at Atlantic City, N. J., June 20, 1957, and at St. Louis, Mo., February 11, 1958. The new officers of the committee are studying the scope of the committee's activities and are considering new areas and additional members to strengthen its coverage.

The brittle fracture problem and crack propagation have been discussed at considerable length. The aircraft industry is in need of a suitable tear test. The brittle fracture field in general needs clarification and unification. To this end a Session on Crack Propagation[1] is being sponsored during the 1958 Annual Meeting at Boston.

Another topic of interest is behavior of material under repeated loading into the plastic range. It has been arranged for L. F. Coffin to give a résumé of work in this field[2] at the Annual Meeting.

The problem of sonic fatigue has been discussed and an attempt will be made to find speakers on this subject.

Other topics discussed were tests of gasket materials, six-component testing, new types of impact testing, formability tests, and high-temperature compression tests. Generally speaking, it is the function of the committee to discuss situations where conventional tests do not give realistic answers.

Respectfully submitted on behalf of the committee,

R. E. Peterson,
Chairman.

J. W. Caum,
Secretary.

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[1] The papers presented at this session appear in this volume, under Technical Papers.

[2] L. F. Coffin, "A Mechanism for Nonpropagating Fatigue Cracks," see p. 570.

REPORT OF COMMITTEE A-1
ON
STEEL*

The steadily increasing use of prestressed concrete has resulted in two specifications covering the steel wire and wire strand. Tentative Specifications for Uncoated Seven-Wire Stress-Relieved Strand for Prestressed Concrete (A 416 - 57 T)[1] were published late in 1957, and proposed Tentative Specifications for Uncoated Stress-Relieved Wire for Prestressed Concrete are appended to this report.[2] Both of these specifications were developed in Subcommittee V.

Eleven specifications covering bar steels have been revised in Subcommittee XV by including all the common requirements in the new Tentative Specifications for General Requirements for Hot-Rolled and Cold-Finished Carbon and Alloy Steel Bars (A 29 - 57 T).[1] The revised specifications as well as Specification A 29 were published late in 1957.

Subcommittee XIX has revised and realigned the specifications covering sheet and strip steel. It has also developed the Tentative Specifications for Carbon Steel Sheets of Flange and Firebox Qualities (A 414 - 57 T)[1] and the proposed Tentative Specifications for Steel Sheets for Porcelain Enameling.[2] The realignment of coverage led to the withdrawal of the Tentative Specifications for Light-Gage Structural Quality Flat Rolled Carbon-Steel Sheets (A 246 - 52 T) and the publication of the Tentative Specifications for Hot-Rolled Carbon Steel Sheets, Commercial Quality (A 415 - 57 T)[1] as well as the proposed Tentative Specifications for Hot-Rolled Carbon Steel Strip of Commercial Quality.[2]

In the field of steel fasteners, Subcommittee XXVI has completely revised its three specifications covering bolting materials. The three proposed revised specifications A 307, A 325, and A 354 are appended to this report.[3]

Three particularly significant specifications have been developed in the field of tubular products. These are appended to this report as the proposed Tentative Specifications for Butt Welds in Still Tubes for Refinery Service,[2] for Seamless and Electric Resistance Welded Low-Alloy Steel Tubes for Economizers,[2] and for Centrifugally Cast Ferritic Alloy Steel Pipe for High-Temperature Service.[2]

The production in this country in 1957 of basic oxygen steel resulted in requests for our steel product specifications to cover it. The process has been approved for pipe produced to Specifications A 53 and A 120 and is being considered for structural steel, concrete reinforcing bars, bar steel, and sheet and strip products.

Another quite significant new document is the Tentative Method for Ultrasonic Testing and Inspection of Turbine and Generator Steel Rotor Forgings

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[*] Sixty-first Annual Meeting of the Society, June 22–27, 1958.

[1] 1957 Supplement to Book of ASTM Standards, Part 1.

[2] The new tentative specifications appear in the 1958 Book of ASTM Standards, Part 1.

[3] The revised specifications appear in the 1958 Book of ASTM Standards, Part 1.

(A 418 – 57 T)[1] developed by a special task group in Subcommittee VI dealing with brittle failure of rotor forgings. This task group sponsored a special meeting for discussion of the subject with delegates to the World Metallurgical Congress at ASTM Headquarters on November 11, 1957. Technical men from Italy, Germany, Great Britain, France, and Japan attended and entered freely into discussions after four technical papers were given by members of the task group. These papers together with two discussions received after the meeting from abroad were published by the Society in a Special Technical Publication, *ASTM STP No. 231.*

Meetings.—Committee A-1 met on June 19, 1957, in Atlantic City, N. J., in conjunction with the 1957 Annual Meeting of the Society. On June 16, 17, and 18 twenty-six subcommittees, sections, and task groups met to prepare reports for committee action. At the Committee A-1 meeting, P. K. Conn, General Electric Co., discussed typical radiation effects on steel products, correlating these data with appropriate ASTM steel specifications. Another talk was given by N. L. Mochel, Westinghouse Electric Corp., outlining that nuclear power will be the power source of the future and that Committee A-1 should be just as interested in standardizing materials for nuclear power plants as it has been for conventional power plants.

Committee A-1 participated in the 1958 Committee Week in St. Louis, Mo. The main committee met on February 12, and 28 subcommittees, sections, and task groups met on February 9, 10, and 11 to prepare proposals for committee action. On February 11, Committee A-1 sponsored a Steel Industry Luncheon with N. P. Veeder, President, Granite City Steel Co., as the featured speaker.

Personnel.—The committee consists of 330 voting members, including 147 producers, 137 consumers, and 46 general interest members.

R. R. Williams, U. S. Steel Corp., has been appointed chairman of Subcommittee XIX on Steel Sheet, replacing E. F Lundeen who has retired from his position with Inland Steel Co. In view of his many years of service to Committee A-1 and his excellent guidance of the work of Subcommittee XIX during his long term of office as chairman of that group, Mr. Lundeen was elected an honorary member of Committee A-1.

R. B. Stampfle, Bethlehem Steel Co., has been appointed as secretary of Subcommittee I on Steel Rails and Accessories. In Subcommittee VIII on Steel Castings, T. W. Merrill, Vanadium Corp. of America, was appointed vice-chairman.

J. L. Menson, Combustion Engineering, Inc., has been appointed for a two-year term as member-at-large of the Advisory Committee. Other men reappointed for two-year terms were as follows:

C. J. Boyle, General Electric Co.
H. F. Brown, Esso Research & Eng. Co.
F. E. Foster, Riley Stoker Corp.
M. D. Helfrick, Western Electric Co.
J. E. Lattan, Taylor Forge and Pipe Works
J. G. Morrow, Steel Company of Canada, Ltd.
M. A. Pinney, The Pennsylvania Railroad Co.
W. S. Scott, Republic Steel Corp.
L. H. Winkler, Bethlehem Steel Co.

The committee records, with great regret, the deaths of three former members of Committee A-1. F. B. Olcott, of the firm of Shirley, Olcott, and Nichols, was a member of Committee A-1 since 1934, serving on Subcommittees II (Structural Steel) and VIII (Steel Castings). J. J. White, chief metallurgical engineer of the Erie Forge and

Steel Corp., had been quite active in Subcommittee VI (Steel Forgings) and Subcommittee XXII (Piping Materials for High Temperature and Subatmospheric Temperature Service). J. H. Romann, consulting engineer, had represented Tube Turns, Inc., on Subcommittees VI and XXII from 1939 till 1943.

Election of Officers.—The officers elected for the ensuing term of two years were as follows:

Chairman, J. J. Kanter.

Producer Vice-Chairman, C. L. Clark.

Producer Vice-Chairman, A. O. Schaefer.

Consumer Vice-Chairman, W. F. Hodges.

Consumer Vice-Chairman, J. L. Menson.

Secretary, H. L. Fry.

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

On August 15, 1957, the Administrative Committee on Standards, acting for the Society, accepted the following recommendations of Committee A-1:

New Tentative Specifications for:

General Requirements for Hot-Rolled and Cold-Finished Carbon and Alloy Steel Bars (A 29 – 57 T).

Revision of Tentative Specifications for:

General Requirements for Delivery of Rolled Structural Steel (A 6 – 56 T),
Hot Rolled Carbon Steel Bars (A 107 – 52a T),
Cold-Finished Carbon-Steel Bars and Shafting (A 108 – 52 T),
Quenched and Tempered Alloy Steel Bars (A 286 – 52 T),
Alloy Steel Bars to End Quench Hardenability Requirements (A 304 – 55 T),
Carbon Steel Bars Subject to Mechanical Property Requirements (A 306 – 55 T),
Stress Relief Annealed Cold Drawn Carbon Steel Bars (A 311 – 52 T),
Quenched and Tempered Carbon-Steel Bars (A 321 – 52 T),
Hot-Rolled Alloy Steel Bars (A 322 – 52 T),
Cold-Finished Alloy Steel Bars (A 331 – 50 T),
Alloy Steel Bars for Nitriding (A 355 – 52 T), and
Cold-Finished Quenched and Tempered Alloy Steel Bars (A 364 – 52 T).

On September 3, 1957, the Administrative Committee on Standards accepted the following recommendations:

New Tentative Specifications for:

Carbon Steel Chain (A 413 – 57 T),
Carbon Steel Sheets of Flange and Firebox Qualities (A 414 – 57 T),
Hot Rolled Carbon Steel Sheets, Commercial Quality (A 415 – 57 T),
Uncoated Seven-Wire Stress-Relieved Strand for Prestressed Concrete (A 416 – 57 T), and
Upholstery Spring Wire for Zig-Zag Type and Square Formed Type and No-Sag Type Spring Units (A 417 – 57 T).

New Tentative Method for:

Ultrasonic Testing and Inspection of Turbine and Generator Steel Rotor Forgings (A 418 – 57 T).

Revision of Tentative Specifications for:

Carbon Steel Axles for Cars and Tenders (A 21 – 57 T),
Alloy-Steel Bolting Materials for High-Temperature Service (A 193 – 56 T),
Carbon-Steel Castings Suitable for Fusion Welding for High-Temperature Service (A 216 – 56 T),
Flat Rolled Carbon Steel Sheets of Structural Quality (A 245 – 52 T),
Carbon and Alloy Steel Forgings for Turbine Rotors and Shafts (A 293 – 55 T),
Alloy-Steel Bolting Materials for Low-Temperature Service (A 320 – 56 T),
Seamless and Welded Steel Pipe for Low-Temperature Service (A 333 – 55 T),
Alloy Steel Seamless Drum Forgings (A 336 – 57 T),
Ferritic and Austenitic Steel Castings for High-Temperature Service (A 351 – 52 T),
Ferritic Steel Castings for Pressure Containing Parts Suitable for Low-Temperature Service (A 352 – 55 T),
Cold-Rolled Carbon Steel Sheets, Special Killed for Miscellaneous Drawn or Severely Formed Parts (A 365 – 53 T),
Cold-Rolled Carbon Steel Sheets, Commercial Quality (A 366 – 53 T), and
Alloy Steel Castings Specially Heat Treated for Pressure Containing Parts Suitable for High-Temperature Service (A 389 – 56 T).

Revision and Reversion to Tentative of Standard Specifications for:

Black and Hot-Dipped Zinc-Coated (Galvanized) Welded and Seamless Steel Pipe for Ordinary Uses (A 120 - 54),
Alloy Steel Castings for Pressure Containing Parts Suitable for High-Temperature Service (A 217 - 55),
Carbon Steel Forgings for Railway Use (A 236 - 57),
Carbon and Alloy Steel Forgings for Magnetic Retaining Rings for Turbine Generators (A 288 - 55),
Carbon and Alloy Steel Forgings for Turbine Generator Rotors and Shafts (A 292 - 55),
Carbon and Alloy Steel Forgings for Turbine Bucket Wheels (A 294 - 55), and
Forged or Rolled Carbon and Alloy Steel Flanges, Forged Fittings, and Valves and Parts for Low-Temperature Service (A 350 - 57).

Withdrawal of Tentative Specifications for:

Light Gage Structural Quality Flat Rolled Carbon-Steel Sheets (A 246 - 52 T).

The new and revised specifications appear in the 1957 Supplement to Book of ASTM Standards, Part 1.

On April 14, 1958, the Administrative Committee on Standards accepted the recommendation to revise the Tentative Specifications for Welded and Seamless Steel Pipe (A 53 - 57 T). The revised specifications have been published as a separate reprint.

RECOMMENDATIONS AFFECTING STANDARDS

The committee is presenting for publication 8 new tentatives and is recommending the revision of 39 tentatives and 14 standards. Three standards are being revised and reverted to tentative status, and a tentative revision of one standard is being recommended. In addition seven standards are being reaffirmed. Eight tentatives are being adopted as standard, of which four are being revised. Three tentatives are recommended for withdrawal.

The standards and tentatives affected, together with the revisions recommended, are given in detail in the Appendix.[4]

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[5]

ACTIVITIES OF SUBCOMMITTEES

Only highlights of the many current subcommittee activities will be covered in the following paragraphs:

There have been many discussions in Subcommittee IX on the possibility of drafting ASTM specifications for mechanical tubing. Because of the wide variety of products included under this category, the task was considered impossible. However in 1958 a task group was appointed to draft two proposed specifications for low-carbon steel mechanical tubing, one to cover the seamless and the other the welded product.

A similar situation has existed with respect to drafting specifications for cold heading wire, since with the wide variety of products covered by this title it seemed impossible to arrive at a proper specification. After some years of investigation, it has finally been decided to have Subcommittee XXVI draft proposed specifications covering commercial round cold-drawn wire for cold heading.

Subcommittee VI has been very active the past few years in the development of a nondestructive test method for inspection of forgings. It is now engaged in writing a test method utilizing dye penetrant and phosphorescent liquid penetration.

Several new specifications are being developed in the field of flat products. A proposed specification for high-strength cold-rolled steel sheets for structural purposes is being drafted by Subcommittee XIX. For pressure vessel construction, Subcommittee XI has been asked to prepare a proposed specification for leaded steel plates for boilers and pressure vessels.

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[4] See p. 100.

[5] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

Finally, in the field of construction and structural work, Subcommittee II is drafting a proposed specification for high-strength steel of the intermediate manganese type for plates, shapes, and bars. Also Subcommittee V has under way proposed specifications for high-strength carbon and alloy billet steels for concrete reinforcement, with a yield strength of 75,000 psi.

This report has been submitted to letter ballot of the committee, which consists of 330 members; 237 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

W. F. COLLINS,
Chairman.

H. L. FRY,
Secretary.

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee A-1 presented to the Society through the Administrative Committee on Standards the following recommendations:

New Tentative Specifications for:

Austenitic Steel Forged and Bored Pipe for High-Temperature Service (A 430 – 58 T), and
High-Strength Billet Steel Bars for Concrete Reinforcement (A 431 – 58 T).

Revision of Tentative Specifications for:

Billet-Steel Bars for Concrete Reinforcement (A 15 – 57 T),
Seamless Carbon-Steel Pipe for High-Temperature Service (A 106 – 55 T),
Cold-Rolled Carbon Steel Strip (A 109 – 58 T),
Forged or Rolled Alloy-Steel Pipe Flanges, Forged Fittings, and Valves and Parts for High-Temperature Service (A 182 – 57 T),
Welded Steel Wire Fabric for Concrete Reinforcement (A 185 – 56 T),
Carbon-Steel Castings Suitable for Fusion Welding for High-Temperature Service (A 216 – 57 T),
Alloy Steel Castings for Pressure Containing Parts Suitable for High-Temperature Service (A 217 – 57 T),
Factory-Made Wrought Carbon Steel and Ferritic Alloy Steel Welding Fittings (A 234 – 58 T),
Flat-Rolled Carbon Steel Sheets of Structural Quality (A 245 – 57 T),
Carbon and Alloy Steel Forgings for Turbine Generator Rotors and Shafts (A 292 – 57 T),
Carbon and Alloy Steel Forgings for Turbine Rotors and Shafts (A 293 – 57 T),
Hot-Rolled Carbon Steel Strip of Structural Quality (A 303 – 58 T),
Alloy-Steel Bolting Materials for Low-Temperature Service (A 320 – 58 T),
Seamless and Welded Steel Pipe for Low-Temperature Service (A 333 – 57 T),
Forged or Rolled Carbon and Alloy Steel Flanges, Forged Fittings, and Valves and Parts for Low-Temperature Service (A 350 – 57 T),
Ferritic and Austenitic Steel Castings for High-Temperature Service (A 351 – 57 T),
Ferritic Steel Castings for Pressure Containing Parts Suitable for Low-Temperature Service (A 352 – 58 T),
Cold-Rolled Carbon Steel Sheets, Special Killed for Miscellaneous Drawn or Severely Formed Parts (A 365 – 57 T),

Cold-Rolled Carbon Steel Sheets, Commercial Quality (A 366 – 57 T),
High Strength Low Alloy Cold-Rolled Steel Sheets and Strip (A 374 – 54 T),
High Strength Low Alloy Hot-Rolled Steel Sheets and Strip (A 375 – 54 T),
Factory-Made Wrought Austenitic Steel Welding Fittings (A 403 – 58 T),
Special Large Size Deformed Billet Steel Bars for Concrete Reinforcement (A 408 – 57 T),
Carbon Steel Sheets of Flange and Firebox Qualities (A 414 – 57 T),
Hot-Rolled Carbon Steel Sheets, Commercial Quality (A 415 – 57 T),
Steel Sheets for Porcelain Enameling (A 424 – 58 T), and
Hot-Rolled Carbon Steel Strip, Commercial Quality (A 425 – 58 T).

Revision and Reversion to Tentative of Standard Specifications for:

Cold-Drawn Steel Wire for Concrete Reinforcement (A 82 – 34),
Carbon Steel Blooms, Billets, and Slabs for Forgings (A 273 – 54), and
Alloy Steel Blooms, Billets, and Slabs for Forgings (A 274 – 54).

These recommendations were accepted by the Society on September 9, 1958, and the new and revised specifications appear in the 1958 Book of ASTM Standards, Part 1.

APPENDIX

PROPOSED RECOMMENDATIONS AFFECTING STANDARDS ON STEEL

In this Appendix, recommendations are given affecting certain specifications both standard and tentative, covering various steel products. The specifications under revision appear in their present form in either the 1955 Book of ASTM Standards, Part 1, or the 1956 or 1957 Supplements to the Book of ASTM Standards, Part 1.

NEW TENTATIVES

The committee recommends for publication as tentative the following specifications as appended hereto:[1]

Tentative Specifications for:

- Uncoated Stress-Relieved Wire for Prestressed Concrete,
- Wrought Alloy Steel Rolls for Cold and Hot Reduction,
- Butt Welds in Still Tubes for Refinery Service,
- Seamless and Electric Resistance Welded Low-Alloy Steel Tubes for Economizers,
- Steel Sheets for Porcelain Enameling,
- Hot-Rolled Carbon Steel Strip of Commercial Quality,
- Centrifugally Cast Ferritic Alloy Steel Pipe for High-Temperature Service, and
- Factory Made Wrought Carbon Steel and Alloy Steel Welding Fittings of Seamless or Welded Construction for Low Temperature Service.

REVISIONS OF TENTATIVES

The committee recommends revisions as indicated of the following tentatives and their continuation as tentative:

Tentative Specification for Steel for Bridges and Buildings (A 7 – 56 T):

Section 4 (b).—In line 7 add the words "basic oxygen" after the words "open hearth."

Section 5 (a).—In the last line revise as indicated above for Section 4 (*b*).

Table II.—In the first column in two places where the words "open hearth or electric furnace" appear, change to read "open hearth, basic oxygen, or electric furnace."

Tentative Specification for General Requirements for Hot Rolled and Cold Finished Carbon and Alloy Steel Bars (A 29 – 57 T):

Section 3 (a).—In the paragraph caption and in the first sentence add the words "basic oxygen" after the words "open hearth."

Section 4 (b).—In the fourth line revise as indicated above for Section 3 (*a*).

Table I.—For phosphorus requirements, in the second column, revise "open hearth steel" to read "open hearth and basic oxygen steel."

Tentative Specifications for Welded and Seamless Steel Pipe (A 53 – 58 T):

Section 4 (a).—Replace the present second sentence with the following: "Samples for chemical analysis shall be taken in accordance with the Method of Sampling Steel, Cast Iron, Open Hearth Iron, and Wrought Iron (ASTM Designation: E 59).[2]"

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[1] The new tentatives appear in the 1958 Book of ASTM Standards, Part 1.

[2] 1958 Book of ASTM Standards, Part 3.

Tentative Specifications for Low-Carbon Steel Track Bolts and Nuts (A 76 - 56 T):

Section 10.—Revise to read as follows:

10. The bolts and nuts shall conform to the plans specified by the purchaser, subject to the following variations (see Note 1):

Shank diameter for cut thread bolt	$+\frac{1}{64}$ in. or $-\frac{1}{32}$ in.
Neck dimensions	$+\frac{1}{32}$ in. or $-\frac{1}{32}$ in.
Length under head	$+\frac{1}{8}$ in. or $-\frac{1}{8}$ in.
Height and diameter of head	$+\frac{1}{16}$ in. or $-\frac{1}{16}$ in.
Nut width	$-(0.05 \times$ thread diameter of bolt) in.
Nut height	$+(0.016 \times$ thread diameter of bolt $+0.012)$ in. or $-(0.016 \times$ thread diameter of bolt $+0.028)$ in.

NOTE 1.—Standard for industrial use is an oval neck bolt (ASA No.: B18.10—1952, p. 7) $\frac{1}{2}$ in., $\frac{5}{8}$ in., $\frac{3}{4}$ in., $\frac{7}{8}$ in., and 1 in. in diameter with a free fit heavy square nut (ASA No.: B18.2—1955, p. 24).

(*a*) The nominal size of bolts shall be the over-all diameter of the threads.

(*b*) The outside diameter of the finished rolled threads shall not exceed the diameter of the shank of a cut thread bolt of corresponding size.

The diameter of the rolled threads shall not exceed the diameter of the shank by more than $\frac{1}{16}$ in. for bolts $\frac{7}{8}$ in. and under in diameter, nor more than $\frac{3}{32}$ in. for bolts 1 in. and over in diameter.

Tentative Specifications for Seamless Steel Boiler Tubes (A 83 - 56 T):

Section 4 (a).—Revise as indicated above for Section 4 (*a*) in Specifications A 53.

Tentative Specifications for Forged or Rolled Steel Pipe Flanges, Forged Fittings, and Valves and Parts for High-Temperature Service (A 105 - 57 T):

Table I.—For both grades add a maximum permissible carbon content of 0.35 per cent to the table and delete the present footnote *a*. Add a new footnote *a*, referenced in the table to the silicon content, to read "It may be necessary to add silicon to the composition for grade II and for the heavier thicknesses of grade I flanges in order to meet the required tensile properties. The silicon content shall not exceed 0.35 per cent."

Section 5 (d).—Revise by the addition of the following words: "except by agreement between the manufacturer and the purchaser."

Tentative Specification for Hot-Rolled Carbon Steel Bars (A 107 - 57 T):

Section 4.—Add the words "basic oxygen" after the words "open hearth."

Table I.—In the first subtitle add the words "basic oxygen" after the words "open hearth."

Also in footnote *a*, in two places, add the words "basic oxygen" after the words "open hearth."

Tentative Specification for Cold-Finished Carbon-Steel Bars and Shafting (A 108 - 57 T):

Section 4.—Add the words "basic oxygen" after the words "open hearth.'

Table I.—Revise the first and second subtitles by adding the words "basic oxygen" after the words "open hearth." Revise footnote *a* as indicated above for Table II in Specification A 107.

Tentative Specifications for Cold-Rolled Carbon Steel Strip (A 109 - 49 T):

These specifications have been extensively revised and are appended hereto in their proposed revised form.[3]

Tentative Specifications for Electric-Resistance-Welded Steel Pipe (A 135 - 57 T):

Section 5 (a).—Add a new second sentence to read "Samples for chemical analysis shall be taken in accordance with the Method of Sampling Steel, Cast Iron, Open-Hearth Iron, and

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[3] The revised tentatives appear in the 1958 Book of ASTM Standards, Part 1.

Wrought Iron (ASTM Designation: E 59).[2]"

Tentative Specifications for Seamless Low-Carbon and Carbon-Molybdenum Steel Still Tubes for Refinery Service (A 161 – 55 T):

Section 1.—Add a new Paragraph (*c*) to read "Where butt welding is used to obtain lengths longer than single lengths normally produced on the mill, the Specifications for Butt Welds in Still Tubes for Refinery Service (ASTM Designation: A 422)[4] shall cover the requirements for butt welds."

Section 6 (*a*).—Revise as indicated above for Section 4 (*a*) in Specifications A 53.

Tentative Specifications for Electric-Resistance-Welded Steel and Open-Hearth Iron Boiler Tubes (A 178 – 56 T):

Section 5 (*a*).—Revise as indicated above for Section 4 (*a*) in Specifications A 53.

Tentative Specifications for Seamless Cold-Drawn Low Carbon Steel Heat Exchanger and Condenser Tubes (A 179 – 56 T):

Section 1 (*b*).—Add a note after this section to read: "NOTE.—Tubing smaller in outside diameter and having thinner wall than indicated herein is available. These smaller sizes may require different mechanical and manipulating tests which should be a matter of agreement between the manufacturer and the purchaser."

Section 5 (*a*).—Revise as indicated above for Section 4 (*a*) in Specifications A 53.

Tentative Specifications for Forged or Rolled Steel Pipe Flanges, Forged Fittings, and Valves and Parts for General Service (A 181 – 57 T):

Table I.—Revise as indicated above for Table I in Specifications A 105.

Tentative Specifications for Seamless Steel Boiler Tubes for High-Pressure Service (A 192 – 57 T):

Section 1 (*b*).—Revise as indicated above for Section 1 (*b*) in Specifications A 179.

Section 5 (*a*).—Revise as indicated above for Section 4 (*a*) in Specifications A 53.

Tentative Specifications for Alloy-Steel Bolting Materials for High-Temperature Service (A 193 – 57 T):

Section 8 (*b*).—Revise to read as follows:

> (*b*) The yield point shall be determined by the drop of the beam or other approved method, as described in Section 7 (*f*) (*1*), (*2*), or (*3*) of Methods A 370. The speed of testing shall not exceed ⅛ in. per min of crosshead speed. Where a definite yield point is not exhibited, the yield strength corresponding to a limiting permanent offset of 0.2 per cent of the gage length of the specimen shall be used as described in Section 7 (*g*) (*1*) of Methods A 370. The tensile strength shall be determined at a maximum crosshead speed of 1½ in. per min.

Section 9.—Revise Paragraph (*a*) to read as follows:

> (*a*) Tension test specimens taken from finished bolts, screws, studs or stud bolts shall be machined to the form and dimensions and shall be taken from positions shown in Section F 2, Paragraph (*d*) of Methods A 370. Tension test specimens from bar stock are covered by Section 5 (*b*) (*2*), (*3*) and (*4*).

Delete the present Paragraphs (*b*) and (*c*) and reletter Paragraph (*d*) as new Paragraph (*b*).

Tentative Specifications for Carbon and Alloy Steel Nuts for Bolts for High-Pressure and High-Temperature Service (A 194 – 57 T):

Section 8 (*c*).—Revise to read as follows:

> (*c*) The hardness may be measured by the Brinell or Rockwell hardness tests. The position of the test and descriptions of the methods are

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[4] 1958 Book of ASTM Standards, Part 1.

given in Section F 5, Paragraph (*b*) and Sections 9 to 11, inclusive, of Methods A 370.

Tentative Specifications for Seamless Cold-Drawn Intermediate Alloy-Steel Heat-Exchanger and Condenser Tubes (A 199 – 56 T):

Section 1 (*a*).—Revise as indicated above for Section 1 (*b*) in Specifications A 179.

Section 6 (*a*).—Replace the present second and third sentences by the new sentence recommended above for Section 4 (*a*) in Specifications A 53.

Tentative Specifications for Seamless Intermediate Alloy-Steel Still Tubes for Refinery Service (A 200 – 55 T):

Section 1.—Add a new Paragraph (*d*) to read as indicated above for new Paragraph (*c*) to Section 1 in Specifications A 161.

Section 6 (*a*).—Revise as indicated above for Section 4 (*a*) in Specifications A 53.

Tentative Specifications for Seamless Carbon-Molybdenum Alloy-Steel Boiler and Superheater Tubes (A 209 – 55 T):

Section 6 (*a*).—Replace the second and third sentences by the new sentence recommended above for Section 4 (*a*) in Specifications A 53.

Tentative Specifications for Medium Carbon Seamless Steel Boiler and Superheater Tubes (A 210 – 55 T):

Section 1 (*b*).—Revise as indicated above for Section 1 (*b*) in Specifications A 179.

Section 4.—Change the permissible maximum manganese content from the present "0.80" to read "0.93" per cent.

Section 6 (*a*).—Replace the second and third sentences by the new sentence recommended above for Section 4 (*a*) in Specifications A 53.

Tentative Specifications for Seamless Alloy Steel Boiler, Superheater, and Heat Exchanger Tubes (A 213 – 57 T):

Section 1.—Add a new Paragraph (*b*) to read as follows, relettering subsequent paragraphs accordingly:

> (*b*) Grade TP 321 H is a modification of grade TP 321 intended for high-temperature service such as for superheaters and reheaters.

Reletter the present Paragraph (*b*) as Paragraph (*c*) and revise as indicated above for Section 1 (*b*) in Specifications A 179.

Section 4.—In Paragraph (*a*) in the first sentence after the words "all material" add the words "except grade TP 321 H."

Add a new Paragraph (*c*) to read as follows:

> (*c*) The heat treatment of grade TP 321 H shall be at a minimum temperature of 2000 F. As evidence that the material has received this treatment it shall exhibit a grain size of No. 7 or coarser as determined in accordance with the classification of Austenite Grain Size in Steels (ASTM Designation: E 19).[2]

Section 7 (*a*).—Revise as indicated above for Section 4 (*a*) in Specifications A 53.

Table II.—Add a new grade TP 321 H with the following chemical requirements:

Carbon, per cent	0.04 to 0.10
Manganese, max, per cent	2.00
Phosphorus, max, per cent	0.030
Sulfur, max, per cent	0.030
Silicon, max, per cent	0.75
Nickel, per cent	9.00 to 13.00
Chromium, per cent	17.00 to 20.00
Titanium, per cent	[a]

[a] Grade TP 321 H shall have a titanium content of not less than four times the carbon content and not more than 0.60 per cent.

In the present footnote *a* of Table II, change the second sentence to read "Small outside diameter are defined as those less than 0.500 in. in outside diameter and light wall tubes as those less than 0.049 in. in average wall thickness (0.040 in. in minimum wall thickness)."

Section 14.—Add a new Paragraph (*g*) to read:

(*g*) Grain size determinations on grade TP 321 H, as specified in Section 4 (*c*), shall be made on the same number of tubes as prescribed for the flattening tests in Paragraph (*d*).

Section 21 (*a*).—Add a new sentence to read "For grade TP 321 H the marking shall also include the heat number and the heat treatment lot identification."

Tentative Specifications for Electric-Resistance-Welded Steel Heat-Exchanger and Condenser Tubes (A 214 - 56 T):

Tentative Specifications for Electric-Resistance-Welded Steel Boiler and Superheater Tubes for High-Pressure Service (A 226 - 57 T):

Section 5 (*a*).—Replace the second and third sentences by the new sentence recommended above for Section 4 (*a*) in Specifications A 53.

Tentative Specifications for Factory-Made Wrought Carbon Steel and Ferritic Alloy Steel Welding Fittings (A 234 - 57 T):

Section 3 (*b*).—Revise to read as follows:

(*b*) Manufacturing operations necessitating seams joined by fusion welding shall comply with the following:

(*1*) Carbon steel fittings having nominal wall thicknesses equal to or less than extra strong IPS are to be (*a*) welded by welders, welding operators, and welding procedures qualified under the provisions of Section IX of the ASME Boiler and Pressure Vessel Code, (*b*) heat treated in accordance with Section 6 of this specification, and (*c*) radiographically examined by selecting a minimum of four fittings out of each 100 fittings or less in each production lot.[a] These fittings shall be selected at random and the welds therein radiographed throughout their entire length in accordance with Paragraph UW-51 of Section VIII of the ASME Boiler and Pressure Vessel Code. If any of these tests indicate substandard welding, two additional fittings shall be radiographed for each defective fitting. If any of these fail to meet the standards, additional fittings shall be examined until the limits of unacceptable welding are determined. However, if any ten of the fittings selected for radiography indicate substandard welding, the entire lot shall be rejected, unless individually approved on the basis of complete radiographic examination.

(*2*) Carbon steel fittings having nominal wall thicknesses heavier than extra strong IPS and all ferritic alloy steel fittings are to be (*a*) welded by welders, welding operators, and welding procedures qualified under the provisions of Section IX of the ASME Boiler and Pressure Vessel Code, (*b*) heat treated in accordance with Section 6 of this specification, and (*c*) radiographically examined throughout the entire length of each weld in accordance with Paragraph UW-51 of Section VIII of the ASME Boiler and Pressure Vessel Code.

(*3*) The joints shall be finished in accordance with the requirements of Paragraph UW-35 (*a*) of Section VIII of the ASME Boiler and Pressure Vessel Code.

Section 6.—Revise to read as follows:

6. (*a*) *Carbon Steel Fittings* (*Grades WPA, WPB, and WPC*):

(*1*) Hot-formed carbon steel fittings upon which the final forming operation is completed at a temperature above 1150 F and below 1800 F need not be heat treated provided they are cooled in still air. If the manufacturer elects to heat treat such fittings it shall be by one of the procedures defined below.

(*2*) Any carbon steel fitting finished at a temperature in excess of 1800 F shall subsequently be normalized, or normalized and tempered.

(*3*) Cold-formed carbon steel fittings, upon which the final forming operation is completed at a temperature below 1150 F shall be stress relieved at 1100 to 1250 F.

(*4*) Carbon steel fittings produced by fusion welding shall be stress relieved following all welding operations at 1100 to 1200 F when the nominal wall thickness at the welded joint is ¾ in. or greater.

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[a] A "lot" shall comprise any sequence of fittings of the same type, size, and thickness, welded under a single procedure.

(*b*) *Alloy Steel Fittings:*

(*1*) Alloy steel fittings of grades WP1 or WP12, whether hot forged or cold formed, as a final heat treatment shall be tempered at not less than 1200 F.

(*2*) Alloy steel fittings of grades WP11, WP22, and WP5 shall be furnished in the full annealed or normalized and tempered condition.

(*3*) Alloy steel fittings produced by fusion welding shall be stress relieved at not less than 1200 F following welding. This applies to all grades of chromium-molybdenum steels in all thicknesses; grade WP1 is required to be stress relieved only when the nominal wall thickness at the welded joint is ½ in. or greater.

(*c*) *Heat Treatment Procedures.*—Fittings after forming at an elevated temperature shall be cooled to a temperature below the critical range under suitable conditions to prevent injuries by too rapid cooling but in no case more rapidly than the cooling rate in still air. Fittings which are to be heat treated shall be treated according to the following procedures:

(*1*) *Full Annealing.*—Fittings shall be uniformly reheated to a temperature above the transformation range and, after being held for a sufficient time at this temperature, cooled slowly to a temperature below the transformation range. Fittings ordinarily are cooled slowly in the furnace but may be transferred and cooled in some other slow cooling medium such as lime.

(*2*) *Normalizing.*—Fittings shall be uniformly reheated to a temperature above the transformation range and subsequently cooled in still air at room temperature.

(*3*) *Tempering* (*Stress Relieving*).—Fittings shall be reheated to a temperature below the transformation range but not less than 1100 F, held at temperature for not less than 1 hr per in. of thickness at the thickest section, and cooled in the furnace or in still air.

New Section.—Add a new Section 8 to read as follows, renumbering the subsequent sections accordingly:

8. *Hardness.*—The hardness of the fittings shall meet the following requirements:

Grade	Brinell Hardness Number, max
WPA, WPB, WPC, WP1, WP11, WP12, and WP22...	201
WP5....................	229

Tentative Specifications for Welded Austenitic Stainless Steel Boiler, Superheater, Heat Exchanger, and Condenser Tubes (A 249 – 57 T):

Section 1 (*b*).—Revise as indicated above for Section 1 (*b*) in Specifications A 249.

Table I.—Revise footnote *a* as indicated above for footnote *a* of Table II in Specifications A 213.

Section 7 (*a*).—Revise as indicated above for Section 4 (*a*) in Specifications A 53.

Tentative Specifications for Electric-Resistance-Welded Carbon-Molybdenum Alloy-Steel Boiler and Superheater Tubes (A 250 – 55 T):

Section 6 (*a*).—Replace the third and fourth sentences with the new sentence recommended above for Section 4 (*a*) in Specifications A 53.

Tentative Specifications for Hot-Rolled Carbon-Steel Strip of Structural Quality (A 303 – 52 T):

Tentative Specifications for Steel Machine Bolts and Nuts and Tap Bolts (A 307 – 55 T):

These specifications have been extensively revised and are appended hereto in their proposed revised form.[3]

Tentative Specifications for Seamless and Welded Austenitic Stainless Steel Pipe (A 312 – 57 T):

Table I.—Revise footnote *a* as indicated above for footnote *a* of Table II in Specifications A 213.

Section 6 (*a*).—Replace the third and fourth sentences with the new sentence recommended above for Section 4 (*a*) in Specifications A 53.

Tentative Specifications for Alloy-Steel Bolting Materials for Low-Temperature Service (A 320 – 57 T):

Section 8 (b).—Revise as indicated above for Section 8 (*b*) in Specifications A 193.

Section 10.—Revise to read as follows:

10. (*a*) Tension test specimens taken from finished bolts, screws, studs, or stud bolts shall be machined to the form and dimensions and shall be taken from positions shown in Section F 2, Paragraph (*d*) of Methods A 370. Tension test specimens from bar stock are covered by Section 5 (*b*) (*2*), (*3*), and (*4*).

(*b*) The impact test specimens shall be tested in accordance with Sections 12 to 14, inclusive, of Methods A 370 using the Charpy keyhole impact specimens described in Fig. 12 or the Charpy U-notch impact specimens described in Fig. 13 of Methods A 370.

(*c*) For sections 1 in. or less in diameter, the test specimens shall be taken at the axis; for sections over 1 to 2 in. in diameter, midway between the axis and the surface; and for sections over 2 in. in diameter, one-quarter of the radius below the surface.

(*d*) The impact test shall be prepared with the notch located on the test specimen surface which is closest to the outside of the section, and parallel to a tangent to the section surface.

Figure 1.—Delete this figure.

Tentative Specifications for Quenched and Tempered Steel Bolts and Studs with Suitable Nuts and Plain Hardened Washers (A 325 – 55 T):

These specifications have been extensively revised and are appended hereto in their proposed revised form.[3]

Tentative Specifications for Seamless and Welded Steel Tubes for Low-Temperature Service (A 334 – 55 T):

Section 1 (a).—Add a new Note 2 at the end of this paragraph to read as follows, renumbering subsequent notes accordingly:

NOTE 2.—Tubing smaller than ½ in. in diameter or with walls thinner than 0.049 in. may require different mechanical and manipulating tests than prescribed in these specifications. Testing of such tubing should be a matter of agreement between the manufacturer and the consumer.

Section 13 (c).—Delete the last sentence.

Tentative Specifications for Ferritic Steel Castings for Pressure Containing Parts Suitable for Low-Temperature Service (A 352 – 57 T):

Table I.—For grade LCB change the maximum permissible manganese content from the present "0.70" to read "1.00" per cent. Delete footnote *a*.

Tentative Specifications for Quenched and Tempered Alloy Steel Bolts and Studs with Suitable Nuts (A 354 – 55 T):

These specifications have been extensively revised and are appended hereto in their proposed revised form.[3]

Tentative Specifications for Heavy-Walled Carbon and Low Alloy Steel Castings for Steam Turbines (A 356 – 57 T):

Section 13 (c).—Revise to read as follows:

(*c*) When dry magnetic particle inspection is used it shall be in accordance with the Method of Dry Powder Magnetic Particle Inspection (ASTM Designation: E 109).[2] Castings showing injurious defects, as judged by the Reference Photographs for Magnetic Particle Indication on Ferrous Castings (ASTM Designation: E 125),[2] shall be rejected or repaired with the approval of the purchaser. The circular or coil method of over-all magnetization or the wet method of magnetic particle inspection, or both, may be used if such methods are more practical.

Tentative Specifications for Structural Steel for Welding (A 373 – 56 T):

Section 3.—Revise to read "The steel shall be made by one or more of the following processes: open hearth, basic oxygen, or electric furnace."

Tentative Specifications for Factory-Made Wrought Austenitic Steel Welding Fittings (A 403 – 56 T):

Section 3 (b).—Revise to read as follows:

(*b*) Fittings containing welded seams or other joining by fusion welding shall be (*a*) welded by welders, welding operators, and welding procedures qualified under the provisions of Section IX of the ASME Boiler and Pressure Vessel Code; (*b*) heat treated in accordance with Paragraph 5 of this specification; and (*c*) radiographically examined throughout the entire length of each weld in accordance with Paragraph UW-51 of Section VIII of the ASME Boiler and Pressure Vessel Code (excepting light-weight fittings covered by Standard Practice SP-43 of the Manufacturers' Standardization Society of the Valve and Fittings Industry).

The joints shall be finished in accordance with the requirements of Paragraph UW-35(*a*) of Section VIII of the ASME Boiler and Pressure Vessel Code.

Tentative Specifications for Welded Large Outside Diameter Light-Wall Austenitic Chromium-Nickel Alloy Steel Pipe for Corrosive or High-Temperature Service (A 409 – 57 T):

Section 3.—Add a new Paragraph (*d*) to read: "(*d*) Samples for chemical analysis shall be taken in accordance with the Method of Sampling Steel, Cast Iron, Open-Hearth Iron, and Wrought Iron (ASTM Designation: E 59).[2]"

TENTATIVE REVISION OF STANDARD

The committee recommends tentative revisions as follows of the Standard Specifications for Carbon Steel Valve Spring Quality Wire **(A 230 – 47)**:

Section 4.—Change the table of chemical requirements to read as follows:

Carbon, per cent	0.60 to 0.75[a]
Manganese, per cent	0.60 to 0.90[a]
Phosphorus, max, per cent	0.025
Sulfur, max, per cent	0.030
Silicon, per cent	0.15 to 0.35

[a] Carbon and manganese may be varied outside the limits of this table by the manufacturer at his discretion, provided the mechanical properties specified are maintained and provided the purchaser does not specifically stipulate otherwise.

Table I.—Change to read as shown in the accompanying Table I.

TABLE I.—TENSILE REQUIREMENTS.

Diameter, in.	Tensile Strength, psi		Reduction of Area, min, per cent
	min	max	
0.093 to 0.128	230 000	250 000	40
0.129 to 0.162	225 000	245 000	40
0.163 to 0.192	220 000	240 000	40
0.193 to 0.225	215 000	235 000	40
0.226 to 0.250	210 000	230 000	40

Section 8.—Replace with the following titled "Coiling and Fracture Test:"

8. (*a*) Wire 0.162 in. and smaller in diameter shall wind on itself as an arbor without breakage. Larger diameter wire up to and including 0.250 in. shall wind without breakage on a mandrel twice the diameter of the wire.

(*b*) The wire shall not show a coarse or cuppy condition when subjected to a fracture test.

Section 10.—Change to read as follows:

10. Transverse sections of the wire properly mounted, polished, and etched shall show no completely decarburized (carbon-free) areas when examined at a magnification of 100 diameters. Partial decarburization shall not exceed a depth of 0.0015 in.

REVISIONS OF STANDARDS AND REVERSION TO TENTATIVE

The committee recommends revisions as indicated of the following standards and their reversion to tentative:

Standard Specifications for Open-Hearth Carbon-Steel Rails (A 1 – 56):

Section 19 (c).—Add the following sentence: "Where the combination of weights or lengths, or both, specified produce more than eight rails ("A" to "H," incl) per ingot, it will be permissible to stamp any additional rails with the letter "H."

With this change the letters "J" and "K" will also be deleted from the present design and size of letters.

Standard Specification for Alloy Steel Forgings for Railway Use (A 238 – 55):

Section 13, Item (3).—Delete the second sentence since it is considered superfluous.

Section 19.—Reletter the present text as Paragraph (*a*) and add a new Paragraph (*b*) to read: "(*b*) The inspector shall examine each forging in each heat for workmanship defects and conformity to the dimensions given on the order or drawing. If in this inspection defects are found which the manufacturer can remedy while the inspector is at the works, he may be allowed to correct such defects."

Standard Methods of Magnetic Particle Testing and Inspection of Heavy Steel Forgings (A 275 – 47):

These methods have been extensively revised and are appended hereto.[5]

ADOPTION OF TENTATIVES AS STANDARD WITHOUT REVISION

The committee recommends that the following tentatives be approved for reference to letter ballot of the Society for adoption as standard without revision:

Tentative Specifications for:

5 per cent Chromium, 0.5 per cent Molybdenum Steel Plates for Boilers and Other Pressure Vessels (**A 357 – 56 T**),
Carbon and Alloy Steel Forgings for Pressure Vessel Shells (**A 372 – 55 T**), and
Chromium-Silicon Steel Spring Wire (**A 401 – 56 T**).

Tentative Recommended Practice for:

Selection of Steel Bar Compositions According to Section (**A 400 – 56 T**).

ADOPTION OF TENTATIVES AS STANDARD WITH REVISION

The committee recommends that the following tentatives be approved for reference to letter ballot of the Society for adoption as standard with revisions as indicated:

Tentative Specifications for Welded and Seamless Open-Hearth Iron Pipe (A 253 – 55 T):

Section 4 (a).—Revise as indicated above for Section 4 (*a*) in Specifications A 53.

Tentative Specifications for Copper Brazed Steel Tubing (A 254 – 55 T):

Section 5 (a).—Revise as indicated above for Section 4 (*a*) in Specifications A 53.

Tentative Specifications for Metal-Arc Welded Steel Pipe for High-Pressure Transmission Service (A 381 – 54 T):

Section 2.—Add a new Paragraph (*e*) to read as indicated above for new Paragraph (*d*) of Section 3 in Specifications A 409.

Tentative Specifications for Alloy Steel Chain (A 391 – 56 T):

Section 7 (c).—Change the fifth sentence to read: "As soon as the beam drops or the gage recedes from ultimate load, the machine is to be stopped and the load reduced to the original seating load."

Section 10.—Delete the word "nominal" in the title and in Paragraph (*a*).

REVISIONS OF STANDARDS, IMMEDIATE ADOPTION

The committee recommends for immediate adoption revisions as indicated of the following standards, and accordingly asks for the necessary nine-tenths affirmative vote at the Annual Meeting in order that the revisions may be referred to letter ballot of the Society:

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[5] The revised methods appear in the 1958 Book of ASTM Standards, Part 3.

Standard Specifications for Mild- to Medium-Strength Carbon-Steel Castings for General Application (A 27 – 57):

Table I.—Add a new grade 70–40 with the following requirements:

Carbon, max, per cent[a]	0.25
Manganese, max, per cent[a]	1.20
Silicon, max, per cent	0.80
Sulfur, max, per cent	0.06
Phosphorus, max, per cent	0.05

Revise the present footnote *a* to read: "For each reduction of 0.01 per cent carbon below the maximum specified, an increase of 0.04 per cent manganese above the maximum specified will be permitted to a maximum of 1.40 per cent for grade 70–40 and 1.00 per cent for the other grades."

Table II.—Add a new grade 70-40 with the following tensile requirements:

Tensile strength, min, psi	70 000
Yield point, min, psi	40 000
Elongation in 2 in. min, per cent	22
Reduction of area, min, per cent	30

Add a new footnote *b* to read: "Grade 70-40 may be used to meet the requirements of grade 70-36, when agreed upon by the manufacturer and purchaser."

Section 14 (c).—Revise as indicated above for Section 14 (*c*) in Tentative Specifications A 356.

Standard Specifications for Structural Steel for Locomotives and Cars (A 113 – 56):

Section 4.—Revise as indicated above for Section 3 in Tentative Specifications A 373.

Standard Specifications for Structural Steel for Ships (A 131 – 56):

Section 3.—Revise as indicated above for Section 3 in Tentative Specifications A 373.

Standard Specifications for Electric-Fusion (Arc)-Welded Steel Pipe (Sizes 4 in. and Over) (A 139 – 55):

Section 5 (a).—Add a new second sentence to read as indicated above for Section 4 (*a*) in Tentative Specifications A 53. The present second sentence will become the third sentence.

Section 11.—Delete Paragraph (*c*), relettering subsequent paragraphs accordingly. Delete the words "and prepared in accordance with Fig. 9" from the present Paragraph (*d*).

Figures 5, 6, 7 and 9.—Delete these figures.

Standard Specifications for Structural Rivet Steel (A 141 – 55):

Section 3.—Revise as indicated above for Section 3 in Tentative Specifications A 373.

Standard Specifications for High-Strength Steel Castings for Structural Purposes (A 148 – 57):

Section 14 (c).—Revise as indicated above for Section 14 (*c*) in Tentative Specifications A 356.

Standard Specifications for One-Wear Wrought Steel Wheels (A 186 – 54):

Section 2.—Revise to read: "The steel shall be made by either or both of the following processes: open hearth or electric furnace."

Standard Specifications for Seamless and Welded Ferritic Stainless Steel Tubing for General Service (A 268 – 57):

Section 1 (a).—Revise as indicated above for Section 1 (*a*) in Tentative Specifications A 334.

Section 7 (a).—Replace the second and third sentences with the new sentence recommended above for Section 4 (*a*) in Tentative Specifications A 53.

Standard Specifications for Seamless and Welded Austenitic Stainless Steel Tubing for General Service (A 269 – 57):

Section 1 (a).—Revise as indicated above for Section 1 (*a*) in Tentative Specifications A 334.

Table I.—Revise footnote *a* as indicated above for footnote *a* of Table II in Tentative Specifications A 213.

Section 7 (a).—Replace the second and third sentences with the new sentence recommended above for Section 4 (*a*) in Tentative Specifications A 53.

Standard Specifications for Seamless and Welded Austenitic Stainless Steel Sanitary Tubing (A 270 – 57):

Section 7 (a).—Replace the second and third sentences with the new sentence as recommended above for Section 4 (*a*) in Tentative Specifications A 53.

Standard Specifications for Seamless Austenitic Chromium-Nickel Steel Still Tubes for Refinery Service (A 271 – 57):

Section 1.—Add a new Paragraph (*d*) to read as indicated above for new Paragraph (*c*) of Section 1 in Tentative Specifications A 161.

Section 7 (a).—Revise as indicated above for Section 4 (*a*) in Tentative Specifications A 53.

Standard Specifications for Low and Intermediate Tensile Strength Carbon-Steel Plates of Structural Quality (Plates 2 in. and Under in Thickness) (A 283 – 54):

Section 3.—Revised as indicated above for Section 3 in Tentative Specifications A 373.

Standard Specifications for Steel Plates for Pressure Vessels for Service at Low Temperatures (A 300 – 57):

Section 5 (b).—Delete the last sentence of this paragraph.

Standard Specifications for Low-Carbon High-Nickel Steel Plate for Pressure Vessels (A 353 – 56):

Section 1.—Revise the first two sentences to read: "This specification covers two grades of low-carbon, high-nickel steel plates of firebox quality, intended primarily for fusion welding, for use in pressure vessels. The maximum thickness of plates to be specified under this specification shall be 2 in. for grade A and 0.580 in. for grade B."

Table I.—Revise to identify the present chemical requirements as those for grade A. Add a new grade B with the same requirements as grade A except the maximum manganese content shall be 0.90 per cent.

Table II.—Revise to identify the present tensile requirements as those for grade A and add the following requirements for grade B:

Tensile strength, min, psi	95 000
Yield strength (0.2 per cent offset), min, psi	65 000
Elongation in 2 in. min, per cent	20.0

Section 10.—Revise to read "As provided in ASTM Specification A 20, each plate shall be legibly stamped or stenciled, depending on the ordered thickness, with the letters "NI" and either "A" or "B" for the applicable grade, in addition to the regular marking."

WITHDRAWAL OF TENTATIVES

The committee recommends the withdrawal of the following tentatives:

Tentative Method of Magnetic Particle Testing and Inspection of Commercial Steel Castings (A 272 – 44 T):

These methods have been replaced by the Tentative Method for Dry Powder Magnetic Particle Inspection (ASTM Designation: E 109).[2]

Tentative Specification for High Tensile Strength Carbon-Manganese Steel Plates for Unfired Pressure Vessels (A 397 – 56 T):

This specification was drafted to replace Case No. 1056 of the ASME Boiler and Pressure Vessel Code. It has been found unacceptable to the Code Committee due to the maximum carbon content being higher than desirable for welding. Committee A-1 has been unable to obtain acceptance of a lower maximum carbon content in the specification. It was felt that to develop the mechanical properties required the greater carbon content was necessary.

Tentative Specifications for Chromium-Silicon Steel Valve Spring Wire (A 402 – 56 T):

Both manufacturers and purchasers have found that this steel does not lend itself to the manufacture of springs requiring high endurance because of an inherent tendency for too great frequency of detrimental defects.

REAPPROVAL OF STANDARDS

The committee recommends the reapproval of the following standards which have stood for six or more years without revision:

Standard Specifications for:

Low Carbon Steel Tie Plates (**A 67 – 48**),
Austenitic Manganese-Steel Castings (**A 128 – 33**),
Hard-Drawn Steel Spring Wire (**A 227 – 47**),
Steel Music Spring Wire (**A 228 – 51**),
Chromium-Vanadium Steel Spring Wire (**A 231 – 41**),
Chromium-Vanadium Steel Valve Spring Quality Wire (**A 232 – 47**), and
Hot-Worked High-Carbon Steel Tie Plates (**A 241 – 48**).

EDITORIAL CHANGES

The committee recommends editorial changes as indicated in the following standards:

Standard Specifications for:

Low-Carbon Steel Joint Bars (**A 3 – 54**),
Medium-Carbon Steel Joint Bars (**A 4 – 54**),
High-Carbon Steel Joint Bars (**A 5 – 54**), and
Quenched Carbon-Steel Joint Bars (**A 49 – 54**).

In the section on "Workmanship" of these four specifications the present requirement is to shear the bars to length. This sentence will be changed to read "The bars shall be either sheared or sawed to length and the punching shall conform to the dimensions specified by the purchaser."

REPORT OF COMMITTEE A-3

ON

CAST IRON*

Committee A-3 on Cast Iron held two meetings during the year: in Atlantic City, N. J., on June 21, 1957, and in St. Louis, Mo., on February 14, 1958. A number of subcommittees met in conjunction with the main committee.

The committee consists of 125 members, of whom 121 are voting members; 43 are classified as producers, 44 as consumers, and 34 as general interest members. There are 4 consulting members.

The officers elected for the ensuing term of two years are as follows:

Chairman, T. E. Eagan.
Vice-Chairman, R. A. Clark.
Secretary, H. W. Lownie, Jr.
Membership Secretary, C. F. Walton.

C. K. Donoho has accepted the chairmanship of Subcommittee 7 on Microstructure of Cast Irons; H. W. Ruf has succeeded T. E. Eagan as chairman of Subcommittee 24 on Nodular Iron; G. P. Krumlauf replaces D. E. Krause as chairman of Subcommittee 25 on Methods of Chill Testing Cast Iron; and W. M. Ruprecht succeeds George Dinges as chairman of Subcommittee 26 on Low Temperature Properties of Cast Iron.

Contact is being maintained by Committee A-3 with Technical Committee 25 on Cast Iron of the International Organization for Standardization (ISO).

A task force has been appointed for the purpose of studying the scope and activities of Committee A-3 and the advisability of reorganization in the interest of increased efficiency.

A Compilation of ASTM Standards on Iron Castings has been issued for the first time. Included are specifications for pig iron, gray iron castings, cast iron pipe, nodular iron castings, and malleable iron castings. General test methods are listed. It is expected the compilation will prove valuable to producers and consumers of the material.

ADOPTION OF TENTATIVES AS STANDARD WITHOUT REVISION

The committee recommends that the following tentatives be approved for reference to letter ballot of the Society for adoption as standard without revision:

Tentative Methods of Chill Testing of Cast Iron (**A 367 – 55 T**),[1] and
Tentative Specification for High-Strength Nodular Iron Castings (**A 396 – 55 T**).[1]

REVISION OF STANDARD, IMMEDIATE ADOPTION

The committee recommends for immediate adoption revisions as follows of the Standard Specifications for Automotive Gray Iron Castings (**A 159 – 55**),[1] and accordingly requests the necessary nine-tenths affirmative vote at the Annual Meeting in order that the revisions may be referred to letter ballot of the Society:

Table II.—Add a third column cover-

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* Sixty-first Annual Meeting of the Society, June 22–27, 1958.

[1] 1955 Book of ASTM Standards, Part 1.

TABLE I.—CHEMICAL, PHYSICAL, AND STRUCTURAL REQUIREMENTS OF AUTOMOTIVE GRAY IRON CASTINGS FOR BRAKE DRUMS AND CLUTCH PLATES.

(Addition to Table II, Specifications A 159 - 55)

	Alloy No. 115[b,e]
CHEMICAL COMPOSITION	
Total carbon, per cent	3.50 min (mandatory)
TYPICAL COMPOSITIONAL RANGES	
Silicon, per cent	1.10 to 1.70 (as desired)
Manganese, per cent	0.60 to 0.90 (as desired)
Sulfur, max, per cent	0.12
Phosphorus, max, per cent	0.15
Alloys	As required
PHYSICAL PROPERTIES	
Brinell hardness number	207 to 269
Transverse strength, min, lb	2800
Deflection, min, in	0.28
Tensile strength, min, psi	40 000
STRUCTURAL PROPERTIES	
Graphite	type A, sizes 3 to 5
Matrix	fine lamellar pearlite with excess constituent, free cementite or free ferrite or both not to exceed approximately 5 per cent.

[e] Extra heavy duty brake drums where both strength and increased resistance to heat checking are required.

ing Alloy No. 115 as indicated in the accompanying Table I.

For Alloys No. 113 and No. 114, change the max per cent sulfur from "0.14" to read "0.12," and change the max per cent phosphorus from "0.20" to read "0.15."

Appendix.—In Paragraph A2, line 2, change "Table III" to read "Tables III and V."

Change Paragraph A3 to read as follows:

A3. *Alloying Elements.*—When alloying elements are specified for Alloy Nos. 110, 111, 120, 121, and 122, Table IV shows the practical range for commercial limits.

Add a new Paragraph A4 as follows, renumbering the present Paragraph A4 as A5:

A4. *Structural Properties of Alloy Nos. 123A, B, and C.*—To insure good wearing characteristics, the microstructure extending 45 deg on both sides of the centerline of the cam nose and to a minimum depth of $\frac{1}{8}$ in. from the surface shall consist of primary carbides (either in the form of acicular, cellular, or a mixture thereof) and types[2] A and E, and size 4 to 7 graphite flakes in a fine pearlite matrix.

The amount of primary carbides required shall be determined by the purchaser and shall be a matter of agreement between the manufacturer and the purchaser.

In a majority of cases the camshaft castings are selectively heat treated by flame or induction hardening of the cam lobes to develop a martensite matrix. The depth of hardened case shall be determined by the purchaser.

Add the following to the end of the present Paragraph A4:

No. 123.—For camshafts.

Add a new Table V as indicated in the accompanying Table II.

REAPPROVAL OF STANDARDS

The committee recommends the reapproval of the following standards which have stood for more than six years without revision:

Standard Specification for Lightweight and Thin-Sectioned Gray Iron Castings (**A 190** - **47**), and

Standard Definitions of Terms Relating to Cast Iron (**A 196** - **47**).

TENTATIVES CONTINUED WITHOUT REVISION

The committee recommends the continuation without revision of the following tentative specifications:

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[2] Recommended Practice for Evaluating the Microstructure of Graphite in Gray Iron (ASTM Designation: A 247), 1958 Book of ASTM Standards, Part 3.

TABLE II.—CHEMICAL, PHYSICAL, AND STRUCTURAL REQUIREMENTS OF AUTOMOTIVE ALLOY GRAY IRON CASTINGS FOR CAMSHAFTS.
(New Table V, Specifications A 159 – 55)

	Alloy 123A	Alloy 123B	Alloy 123C
TYPICAL COMPOSITIONAL RANGES			
Total carbon, per cent	3.10 to 3.40	3.10 to 3.45	3.40 to 3.75
Silicon, per cent	2.10 to 2.40	2.10 to 2.40	2.10 to 2.35
Manganese, per cent	0.50 to 0.80	0.60 to 0.90	0.60 to 0.90
Phosphorus, per cent	0.20 max	0.20 max	0.15 max
Sulfur, per cent	0.15 max	0.20 max	0.15 max
Chromium, per cent	0.80 to 1.10	0.85 to 1.20	1.00 to 1.25
Molybdenum, per cent	0.40 to 0.60	0.40 to 0.60	0.50 to 0.70
Nickel, per cent	...	0.20 to 0.45	...
Copper, per cent	...	...	1.40 to 1.70
PHYSICAL PROPERTIES			
Brinell hardness number[a]	248 to 311	...	...
Transverse strength, min, lb.[b]	2600	...	...
Deflection, min, in.[b]	0.27	...	...
Tensile strength, min, psi[b]	40 000	...	...

[a] The casting hardness shall conform to the same hardness range as specified for the test bar and shall be taken on a bearing surface as agreed upon between the manufacturer and the purchaser.

[b] Properties determined on 1.2 in. diameter test bar broken on 18-in. centers.

Tentative Specifications for:

Foundry Pig Iron (**A 43 – 55 T**),

Chilled and White Iron Castings (**A 360 – 55 T**), and

Cast Nodular Iron for Pressure Containing Parts for Use at Elevated Temperatures (**A 395 – 56 T**).

Experience with these new specifications is still too limited to warrant either revision or advancement to standard at this time.

EDITORIAL CHANGE

The committee recommends editorial changes as follows of the Standard Specifications for Gray Iron Castings (**A 48 – 56**):[3]

Figure 2.—Replace with Fig. 16 of Methods of Tension Testing of Metallic Materials (E 8 – 57 T).

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[4]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee 6 on General Castings (H. W. Lownie, Jr., chairman) anticipates major revisions of Specification A 48 in the near future. Data on the properties of iron cast into various types of specimens are being assembled with this in view. Specimens other than the present standard arbitration type used by various foundries are being investigated.

Subcommittee 12 on Austenitic Cast Iron (Hugo Larson, chairman) is completing specifications covering the various grades of austenitic iron castings.

Subcommittee 22 on Elevated Temperature Properties of Cast Iron (J. S. Vanick, chairman).—Progress on Research Project SP-2 of the Joint ASTM-ASME

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[3] 1956 Supplement to Book of ASTM Standards, Part 1.

[4] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

Committee on Effect of Temperature on the Properties should be completed and reported on at the June, 1958 meeting. This project deals with elevated temperature properties of commercial irons of the gray iron and nodular graphite types.

This report has been submitted to letter ballot of the committee, which consists of 121 voting members; 95 members returned their ballots, of whom 91 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

D. E. KRAUSE,
Chairman.

R. A. CLARK,
Secretary.

REPORT OF COMMITTEE A-5

ON

CORROSION OF IRON AND STEEL*

Committee A-5 on Corrosion of Iron and Steel held two meetings during the year: on June 21, 1957, at Atlantic City, N. J., and on February 14, 1958, at St. Louis, Mo.

The committee consists of 110 members, of whom 52 are classified as producers, 34 as consumers, and 24 as general interest members.

The officers elected for the ensuing term of two years are as follows:

Chairman, H. F. Hormann.

First Vice-Chairman, E. K. Camp.

Second Vice-Chairman, J. F. Occasione.

Secretary, C. P. Larrabee.

The Fiftieth Anniversary of Committee A-5 was commemorated by the publication of the high points of its history in the February, 1958, issue of the ASTM BULLETIN.

T. R. Galloway and E. K. Camp reported to the committee on nuclear problems as they may bear on the corrosion of carbon steel.

AMERICAN STANDARDS

The following standards, under the jurisdiction of Committee A-5, were approved by the American Standards Assn. as American Standard on January 23, 1958:

Standard Specifications for:

Zinc-Coated (Galvanized) Iron or Steel Farm-Field and Railroad Right-of-Way Wire Fencing (ASTM A 116 - 57; ASA G8.9—1958).

Zinc-Coated (Galvanized) Iron or Steel Barbed Wire (ASTM A 121 - 57; ASA G8.10—1958)

NEW TENTATIVE

The committee recommends for publication as tentative the Methods of Test for Weight of Coating on Aluminum-Coated Iron or Steel Articles as appended hereto.[1]

REVISION OF TENTATIVES

The committee recommends revisions as indicated of the following tentative specifications and their continuation as tentative:

Specifications for Zinc-Coated (Galvanized) Iron or Steel Sheets, Coils, and Cut Lengths (A 93 - 55 T):[2]

Section 6 (c).—Change to read as follows:

> (*c*) The minimum check limit by the single-spot test shall be that one of the three specimens of the triple-spot test bearing the lightest coating, or the purchaser may select a single specimen taken from any part of a test sheet. For material narrower than 2.25 in., test specimens shall be of such a length as to render the area of the specimen equal to 5.06 sq in.

Section 10 (b).—Delete the second sentence.

Section 11 (b).—Change the size of lot to read 30,000-lb lot.

Section 19.—Change the last sentence to read, "Unless otherwise specified, all inspections and tests (except check analysis) shall be made at the manufacturer's works prior to shipment. Such sampling and inspection shall be made in conjunction with, and to the extent of, the manufacturer's regular inspection operations unless otherwise specified."

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* Sixty-first Annual Meeting of the Society, June 22-27, 1958.

[1] The new tentative method appears in the 1958 Book of ASTM Standards, Part 3.

[2] 1955 Book of ASTM Standards, Part 1.

Specifications for Long Terne Iron or Steel Sheets, Coils and Cut Lengths (A 308 - 53 T):[2]

Revise as appended hereto.[3]

Specifications for 1.25-oz Class Coating (Pot Yield) Zinc-Coated (Galvanized) Iron or Steel Roofing Sheets (A 361 - 55 T):[2]

Section 15.—Change the last sentence to read, "Unless otherwise specified, all inspection and tests (except check analysis) shall be made at the manufacturer's works prior to shipment. Such sampling and inspection shall be made in conjunction with and to the extent of the manufacturer's regular inspection operations unless otherwise specified."

ADOPTION OF TENTATIVE AS STANDARD WITH REVISION

The committee recommends that the Tentative Specification for Tie Wires, Zinc-Coated (Galvanized) Steel (**A 112 - 55 T**)[2] be approved for reference to letter ballot of the Society for adoption as standard with revisions as indicated.

Section 7.—In line 5, change "0.083 in. in diameter" to read, "0.095 in. in diameter and smaller."

Section 13.—Add a new wire size having a nominal diameter of 0.095 in. and a Bwg size of 13.

ADOPTION OF TENTATIVES AS STANDARD WITHOUT REVISION

The committee recommends that the following tentatives be approved for reference to letter ballot of the Society for adoption as standard without revision:

Tentative Specifications for:

Zinc-Coated Steel Wire Strand ("Galvanized" and Class A "Extra Galvanized") (**A 122-54 T**)[2]

Zinc-Coated Steel Wire Strand (Class B and Class C Coatings) (**A 218 - 54 T**)[2]

Wire Strand, Overhead Ground, Zinc-Coated Steel (**A 363 - 55 T**)[2]

Poultry Netting (Hexagonal and Straight Line) and Woven Poultry Fencing, Zinc-Coated (Galvanized) Steel (**A 390 - 56 T**)[4]

REVISION OF STANDARD, IMMEDIATE ADOPTION

The committee, jointly with Committee B-8 on Electrodeposited Metallic Coatings, recommends revisions for immediate adoption in the Standard Methods of Test for Local Thickness of Electrodeposited Coatings (**A 219 - 54**).[2] These revisions are set forth in the Report of Committee B-8.

REAPPROVAL OF STANDARDS

The committee recommends reapproval of the following standards which have stood for six years without revision:

Standard Specifications for:

Line Wire, Zinc-Coated (Galvanized) "Iron" Telephone and Telegraph (**A 111 - 52**).

Line Wire, Zinc-Coated (Galvanized) High Tensile Steel Telephone and Telegraph (**A 326 - 52**).

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[5]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee VII on Methods of Testing (O. B. Ellis, chairman) prepared the method of test for weight of coatings on hot-dipped aluminum-coated iron and steel referred to earlier in the report. It concurred in the revision of Methods

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[3] The revised tentative appears in the 1958 Book of ASTM Standards, Part 1.

[4] 1956 Supplement to Book of ASTM Standards, Part 1.

[5] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

A 219 - 54 suggested by Committee B-8. The report of the task group on Magnetic Coating Measurements (V. V. Kendall, chairman) is expected in the near future.

Subcommittee XI on Sheet Specifications (E. P. Beachum, chairman) recommended the revision of Tentative Specifications A 93 - 54 T, A 308 - 53 T and A 361 - 55 T referred to earlier in this report. The subcommittee plans to prepare specifications for galvanized sheets of structural quality and for galvanized sheets for culverts, underdrains, and arches.

Subcommittee XII on Wire Specifications (J. F. Occasione, chairman).—Criticisms of the Tentative Specification for Zinc-Coated Steel Chain-Link Fence Fabric (A 392 - 55 T) have not as yet been resolved and further consideration is needed. Additional data are being gathered for a specification for flat armor tape. The subcommittee recommended the adoption as standard of Tentative Specifications A 112 - 55 T, A 363 - 55 T, A 390 - 56 T, and the reapproval of Standard Specifications A 111 - 52 and A 326 - 52.

Subcommittee XIII on Hardware Specifications (B. J. Barmack, chairman) is reviewing Tentative Specifications A 384 - 55 T, A 385 - 55 T, and A 386 - 55 T for the purpose of adoption as standard. Specification A 123 - 53 for Zinc (Hot-Galvanized) Coatings on Products Fabricated from Rolled, Pressed, and Forged Steel Shapes, Plates, Bars and Strip is being studied with a view of raising the present average weight of coating of 2 oz per sq ft of surface, and of raising the present minimum value of 1.8 oz. Specification A 153 - 53 for Zinc Coating (Hot-Dip) on Iron and Steel Hardware is being studied for the purpose of raising the average and minimum values for certain classes of materials. The use of magnetic thickness gages will be recommended to replace the Preece test which was deleted from Specification A 153. Recommended Practice for Safeguarding Against Embrittlement (A 143 - 46) is being revised to emphasize precautions in shop practice in punching holes. Other subjects under consideration include the hammer test, and inspection of galvanizing and regalvanizing specifications for hardware.

Subcommittee XIV on Sheet Tests (C. P. Larrabee, chairman) inspected the atmospheric exposure tests of corrugated sheets at Altoona and at State College, Pa., on April 24, 1957. The report of each inspection follows:

Altoona, Pa.—In 1954 it was reported that only two 22-gage sheets were unperforated. Originally both had 2.5 oz per sq ft of zinc coating and were identified as "T" and "K." At the 1955 inspection it was found that the "T" sheet had been "damaged mechanically"; therefore it was decided that further data would not be recorded. The unperforated "K" sheet was moved to the roof of the Pennsylvania Railroad laboratory; at the 1957 inspection no holes were visible. When this sheet is found to be perforated the test will be complete, and a table with the final results will be published.

State College, Pa.—The present status of this test is shown in Table I. No new perforations of uncoated sheets were found, nor has a perforation been found in any galvanized sheet.

Subcommittee XV on Wire Tests (F. M. Reinhart, chairman) inspected the wire specimens undergoing atmospheric exposure tests at Bridgeport, Conn., and at State College, Pa., during April, 1957. Wire exposure tests at other locations were inspected by the university people in charge of the various sites during the Fall of 1957. Because of the amount of work connected with the 20-Year Wire Report which is in process of prepara-

TABLE I.—LIFE OF CORRUGATED GALVANIZED AND BLACK SHEETS, 26 BY 30 IN.
Exposed at State College, Pa., on April 23, 1926.

Sheet[b]	No. 16 Gage, 2.5 oz per sq ft[a]		No. 22 Gage, 2.5 oz per sq ft[a]			No. 22 Gage, 2.0 oz per sq ft[a]			No. 22 Gage, 1.5 oz per sq ft[a]			No. 22 Gage, 1.25 oz per sq ft[a]			No. 22 Gage, 0.75 oz per sq ft[a]			No. 28 Gage, 0.75 oz per sq ft[a]			No. 22 Gage Uncoated
	Years to		Years to			Years to			Years to			Years to			Years to			Years to			
	1st Rust	100 per cent Rust	1st Rust	100 per cent Rust	Perforation	1st Rust	100 per cent Rust	Perforation	1st Rust	100 per cent Rust	Perforation	1st Rust	100 per cent Rust	Perforation	1st Rust	100 per cent Rust	Perforation	1st Rust	100 per cent Rust	Perforation	Years to Perforation
R (T)	24.0	*	26.0	*	*	19.0	*	*	17.5	*	*	14.5	*	*	[c]			[c]			26.0
(B)	27.0	*	29.0	*	*	20.0	*	*	16.5	*	*	15.0	*	*	[c]			[c]			
V (T)	27.0	*	21.5	*	*	18.5	*	*	16.0	*	*	15.0	*	*	9.5	23.0	*	[c]			24.0(V)
(or U) (B)	24.0	*	29.0	*	*	25.0	*	*	15.5	*	*	16.5	*	*	10.5	24.0	*	[c]			25.0(U)
N (T)	[c]		20.5	*	*	22.5	*	*	17.5	*	*	12.0	*	*	6.5	20.5	*	11.0	23.0	*	28.0
(B)	[c]		28.0	*	*	22.5	*	*	15.5	*	*	13.5	*	*	9.0	23.0	*	11.5	30.0	[d]	
T (T)	27.0	*	*	*	*	21.0	*	*	16.0	*	*	15.0	*	*	9.0	26.0	*	9.0	24.0	*	*
(B)	31.0	*	*	*	*	23.0	*	*	14.5	*	*	13.5	*	*	10.5	22.5	*	12.0	25.0	*	
Y (T)	21.5	*	22.5	*	*	19.5	*	*	17.5	*	*	14.5	*	*	10.0	22.5	*	9.5	24.0	*	25.0
(B)	22.5	*	29.0	*	*	19.0	*	*	18.5	*	*	14.5	29.0	*	10.5	30.0	*	13.0	26.0	*	
S (T)	22.0	*	28.0	*	*	25.0	*	*	19.0	*	*	14.5	*	*	10.5	21.5	*	11.5	22.0	*	*
(B)	22.5	*	*	*	*	28.0	*	*	19.5	*	*	15.5	30.0	*	11.0	21.5	*	11.5	21.5	*	
C (T)	[c]		22.5	*	*	17.5	*	*	16.0	*	*	14.5	*	*	9.5	24.0	*	[c]			*
(B)	[c]		*	*	*	21.5	*	*	18.5	*	*	14.5	30.0	*	10.0	24.0	*	[c]			
K (T)	23.0	*	25.0	*	*	22.5	*	*	17.5	*	*	14.5	31.0	*	11.5	24.0	*	10.5	23.0	*	*
(B)	24.0	*	30.0	*	*	28.0	*	*	21.0	*	*	16.5	*	*	11.5	22.5	*	11.5	21.0	*	
W (T)	[c]		29.0	*	*	25.0	*	*	16.5	*	*	13.5	31.0	*	[c]			[c]			27.0
(B)	[c]		*	*	*	28.0	*	*	15.0	*	*	14.5	30.0	*	[c]			[c]			
Average	24.6	...	...	...	...	22.5	...	...	17.1	...	...	14.6			10.0	23.5		11.1	24.0		

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* Data not yet available.

[a] Coating weight is "oz per sq ft" of sheet (both sides).

[b] (T) = Corrugated sample from top of sheet on west side of rack with bottom surface upward.
(B) = Corrugated sample from bottom of sheet on east side of rack with top surface upward.

[c] There were no specimens exposed.

[d] Reported as perforation rather than 100 per cent rust in 1956 *Proceedings*.

tion, no report was prepared on the 1957 wire inspections. The 20-Year Wire Report will show as much data as possible in graph form.

Subcommittee XVI on Hardware Tests (A. Mendizza, chairman) has nearly completed the specimen preparation phase of the hardware test program. The thickness of coating of individual specimens is being determined by nondestructive methods, spot checked by stripping methods. Approximately 800 specimens will be placed on the test racks within the next three months. These samples comprise hot-dip zinc and electrodeposited zinc, hot-dip aluminum from two sources, sprayed zinc and sprayed aluminum coatings on carbon steel, four low-alloy steels, and two varieties of nodular iron. They will be exposed at one industrial location (Newark, N. J.) and at two marine environments (80-ft and 800-ft sites at Kure Beach, N. C.). Test specimens include flat and S-shaped panels and round rods.

This report has been submitted to letter ballot of the committee, which consists of 110 members; 90 members returned their ballots, of whom 81 have voted affirmatively, and 0 negatively.

Respectfully submitted on behalf of the committee,

MARC DARRIN,
Chairman.

C. P. LARRABEE,
Secretary.

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee A-5 presented to the Society through the Administrative Committee on Standards the following recommendations:

Revision of Tentative Specifications for:

Zinc-Coated (Galvanized) Iron or Steel Sheets, Coils, and Cut Lengths (A 93 - 58 T),
Long Terne Iron or Steel Sheets, Coils, and Cut Lengths (A 308 - 58 T), and
1.25-oz Class Coating (Pot Yield) Zinc-Coated (Galvanized) Iron or Steel Roofing Sheets (A 361 - 58 T).

These recommendations were accepted by the Standards Committee on September 9, 1958, and the revised tentative specifications appear in the 1958 Book of ASTM Standards, Part 1.

REPORT OF COMMITTEE A-6
ON
MAGNETIC PROPERTIES*

Committee A-6 on Magnetic Properties has held four general meetings since the last published report of the committee as follows: June 28, 1955, at Atlantic City, N. J.; June 18, 1956, at Atlantic City, N.J.; June 21, 1957, at Atlantic City, N.J.; and a meeting on February 12 and 13, 1958, at St. Louis, Mo. during ASTM Committee Week.

There have been a number of changes in membership. It is with sorrow that we record the passing of two faithful committee members whose work has meant a great deal to the Society. Mr. C. J. Witsberger, who was chairman of the Working Group which prepared Specification A 345 – 55, the Standard Specification for Flat Rolled Electrical Steel, passed away May 29, 1955. Mr. Frank P. Fahy, who had been active in the committee since 1917 and is well known as the inventor of the Fahy permeameter, passed away on March 25, 1956.

The basic organization of the committee remains the same as it was at the time of the last published report, June 1955. The representation of Committee A-6 on other ASTM committees and on committees of other standardizing organizations is as follows:

ASTM Committee E-8 on Nomenclature and Definitions, I. L. Cooter.

ASTM Committee E-11 on Quality Control of Materials, K. V. Merrill.

ASTM Committee C-21 on Ceramic Whitewares and Related Products, Subcommittee II on Tests and Specifications, Subsection A-5 on Ferrites, A. C. Beiler.

American Standards Assn., Sectional Committee C42 on Definitions of Electrical Terms, I. L. Cooter.

American Standards Assn., Sectional Committee C61 on Electric and Magnetic Magnitudes and Units, I. L. Cooter.

International Electrotechnical Commission, Subcommittee 14-A on Magnetic Steel, Technical Adviser to U. S. National Committee, A. C. Beiler.

American Society of Tool Engineers, Project on Metal Stamping, G. H. Cole.

The officers elected for the ensuing term of two years are as follows:

Chairman, A. C. Beiler.
Vice-Chairman, D. C. Dieterly.
Secretary, W. S. Eberly.

NEW STANDARD

The committee recommends for immediate adoption as standard the proposed Methods of Test for Alternating Current Magnetic Properties of Laminated Core Specimens as appended hereto,[1] and accordingly asks for the necessary nine-tenths affirmative vote at the Annual Meeting in order that the standard may be referred to letter ballot of the Society.

REVISIONS OF STANDARD, IMMEDIATE ADOPTION

The committee recommends for immediate adoption revisions as appended hereto[2] of the Standard Methods of Test for Alternating Current Core Loss, and Permeability of Magnetic Materials (**A 343 – 54**),[3] and accordingly asks for the necessary nine-tenths affirmative vote at the Annual Meeting in order that the revisions may be referred to letter ballot of the Society.

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* Sixty-first Annual Meeting of the Society, June 22–27, 1958.

[1] The revised tentative appears in the 1958 Book of ASTM Standards, Part 3.

REAPPROVAL OF STANDARDS

The committee recommends the reapproval of the following standards which have stood for six or more years without revision:

Standard Definitions of:

Terms, with Symbols, Relating to Magnetic Testing (**A 340 – 49**), and

Standard Methods of Test for:

Electrical and Mechanical Properties of Magnetic Materials (**A 344 – 52**).

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[4]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Nomenclature and Definitions (I. L. Cooter, chairman) has been revising the magnetic definitions to maintain uniformity between ASTM and the American Standard sponsored by ASA Sectional Committee C 42. This will result in a major change of Definitions of Terms, with Symbols, Relating to Magnetic Testing (A 340 – 49) which should be ready in 1959. Additional work on Interlaminar Resistance Test Methods will undoubtedly result in a major revision of Methods A 344 – 52, which should be available later in 1958 for inclusion in the 1959 revisions. A task group has been at work on Methods of Test of Feebly Magnetic Materials, and considerable work has been done to establish the validity of the new Naval Ordnance Laboratory test procedure.

Three task groups recently activated are as follows: (1) Task Group on Standard Methods of Test Cores for Magnetic Amplifiers or Saturable Reactor Devices, under the chairmanship of W. T. Mitchell; (2) Task Group on the Effect of Coatings, Aluminum Additions, and Other Factors on the Values of Physical Density of Electrical Steels, with D. A. Jones as chairman; and (3) Task Group on Methods of Test for Very High Coercive Force Permanent Magnets, with C. A. Maynard as temporary chairman.

Subcommittee II on Methods of Sampling and Test (J. W. Hale, chairman).—During the past year, there has been activity in several of the task groups under the chairmanship of D. C. Dieterly, in cooperation with the Editorial Subcommittee under the chairmanship of C. C. Horstman, which has resulted in the revision of the Standard Methods of Test for Alternating Current Core Loss, and Permeability of Magnetic Materials (A 343 – 54) and in the proposed Tentative Methods of Test for Alternating-Current Magnetic Properties of Laminated Core Specimens.

Subcommittee II is also working on a revision of the Lamination Factor Test which is a part of Methods A 344.

This report has been submitted to letter ballot of the committee, which consists of 29 voting members; 19 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

A. C. BEILER,
Chairman.

W. S. EBERLY,
Secretary.

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[2] This revision was not published since the committee found it necessary to make a further complete revision of the specification, which will be submitted to the Society within the year. A copy of the completely revised methods under consideration will be furnished on request.

[3] 1955 Book of ASTM Standards, Part 1.

[4] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

REPORT OF COMMITTEE A-7

ON

MALLEABLE IRON CASTINGS*

Committee A-7 on Malleable Iron Castings held two meetings during the year: at Atlantic City, N. J., on June 18, 1957, and at St. Louis, Mo., on February 11, 1958.

Preceding its first meeting the committee had lost by death its beloved, valued, and highly respected Chairman for the past eleven years, William A. Kennedy. He received posthumously the Award of Merit of the Society. The committee also lost with regret the valued services of Charles A. Gutenkunst, Jr., President of Milwaukee Malleable and Gray Iron Works, and a long-time member of Committee A-7.

Following the loss of its chairman, in accordance with the by-laws of the committee, the Advisory Committee appointed officers to serve for the unexpired term. Vice-Chairman Albrecht was advanced to the office of Chairman and L. C. Marshall was named Vice-Chairman.

At the February meeting, completion of plans was announced for mounting 1620 standard malleable, pearlitic malleable, steel, and nodular iron corrosion test plates in May of 1958, at five Corrosion Exposure Test Sites.

A paper by Dr. L. C. Marshall on "Elevated Temperature Properties of Malleable Irons" is to be presented at the Annual Meeting of the Society, June 1958.

The committee consists of 50 members, of whom 24 are classified as producers, 13 as consumers, and 13 as general interest members.

The officers elected for the ensuing term of two years are as follows:

Chairman, W. M. Albrecht.
Vice-Chairman, L. C. Marshall.
Secretary, James H. Lansing.
Advisory Committee: V. A. Crosby and R. V. Osborne.

TENTATIVE CONTINUED WITHOUT REVISION

The committee recommends the continuation without revision of the Tentative Specification for Pearlitic Malleable Iron Castings (**A 220 – 55 T**).[1]

REAPPROVAL OF STANDARDS

The committee recommends the reapproval of the following standards:

Specifications for:
Malleable Iron Castings (**A 47 – 52**),
Cupola Malleable Iron (**A 197 – 47**), and
Malleable Iron Flanges, Pipe Fittings, and Valve Parts for Railroad, Marine, and Other Heavy Duty Service (**A 338 – 54**).

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[2]

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* Sixty-first Annual Meeting of the Society, June 22–27, 1958.

[1] 1955 Book of ASTM Standards, Part 1.

[2] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee VI on Elevated Temperature Properties of Malleable Irons (L. C. Marshall, chairman) was established at the February meeting by vote of the committee, on the recommendation of Chairman Albrecht. This subcommittee will assist its chairman in the continuation of his work on this subject.

This report has been submitted to letter ballot of the committee, which consists of 50 members; 34 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

W. M. ALBRECHT,
Chairman.

JAMES H. LANSING,
Secretary.

REPORT OF COMMITTEE A-10
ON
IRON-CHROMIUM, IRON-CHROMIUM-NICKEL, AND RELATED ALLOYS*

Committee A-10 on Iron-Chrominum, Iron-Chromium-Nickel, and Related Alloys held two meetings during the year: in Atlantic City, N. J., on June 20, 1957 during the Annual Meeting of the Society, and on February 12, 1958 at St. Louis, Mo. during ASTM Committee Week.

The committee consists of 103 members, of whom 45 are classified as producers, 39 as consumers, and 19 as general interest members.

The officers elected for the ensuing term of two years are as follows:

Honorary Chairman, Jerome Strauss.
Chairman, L. L. Wyman.
Vice-Chairman, M. A. Cordovi.
Secretary, L. B. Fonda.

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1957 Annual Meeting, Committee A-10 presented to the Society through the Administrative Committee on Standards the following recommendations:

Revision and Reversion to Tentative of Standard Specifications for:

Corrosion-Resisting Plate, Sheet, and Strip (A 240 - 54), and
Corrosion-Resisting Steel Bars (A 276 - 55).

These recommendations were accepted by the Standards Committee on November 1, 1957, and the revised specifications appear in the 1957 Supplement to Book of ASTM Standards, Part 1.

NEW TENTATIVE

The committee recommends for publication as tentative the Specification for Hot-Rolled and Cold-Finished Corrosion-Resisting Chromium-Nickel-Manganese Steel (Types 201 and 202) Bars, as appended hereto.[1]

REVISION OF TENTATIVE

The committee recommends revision as follows of the Tentative Specifications for Corrosion-Resisting Chromium and Chromium-Nickel Steel Plate, Sheet, and Strip for Fusion-Welded Unfired Pressure Vessels (**A 240 - 57 T**):[2]

Section 8 (*b*).—Change to read as follows:

> (*b*) Between yield and fracture of the test specimen, the tests shall be conducted at a constant strain rate between ⅛ in. per in. per min and ½ in. per in. per min, inclusive, or at a crosshead speed which will give a strain rate within this range. For the purposes of these specifications, the rate of strain may be determined by a strain rate pacer, indicator, or controller, or by dividing the unit elongation by the elapsed time from yield strength to fracture.

REVISION OF STANDARDS, IMMEDIATE ADOPTION

The committee recommends for immediate adoption revisions as indicated of the following standards, and accordingly asks for the necessary nine-tenths affirmative vote at the Annual Meeting in order that the revisions may be referred to letter ballot of the Society:

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* Sixty-first Annual Meeting of the Society, June 22–27, 1958.

[1] The new tentative appears in the 1958 Book of ASTM Standards, Part 1.

[2] 1957 Supplement to Book of ASTM Standards, Part 1.

Standard Specifications for Corrosion-Resisting Chromium-Nickel Steel Plate, Sheet, and Strip (A 167 – 54):[3]

Section 7 (*c*).—Change as indicated above for Section 8 (*b*) of Specifications A 240.

Standard Specifications for High-Strength Corrosion-Resisting Chromium-Nickel Steel Sheet and Strip (A 177 – 54):[3]

Section 8(*c*).—Change as indicated above for Section 8 (*b*) of Specifications A 240.

Standard Specifications for Corrosion-Resisting Steel Billets and Bars for Reforging (A 314 – 55):[3] Revise as appended hereto.[4]

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[5]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee IV on Methods of Corrosion Testing (O. B. Ellis, chairman) is preparing summaries of the stress-corrosion cracking data for publication. Most of the stainless steel specimens for the atmospheric corrosion tests have been supplied. These will be exposed this summer at State College, Pa., Kure Beach, N. C., Newark, N. J., Columbus, Ohio, and Freeport, Tex.

A new task group will be appointed to study a new test for determining intergranular corrosion characteristics of sensitized stainless steels.

Subcommittee V on Mechanical Tests (R. H. Heyer, chairman) has approved a new speed of testing procedure for the stainless steel flat products under the jurisdiction of Committee A-10 which is being proposed for adoption, as mentioned earlier in this report.

Subcommittee VI on Metallography (R. Franks, chairman) is continuing to investigate metallographically the causes of cracking in heavy type 347 stainless steel piping. The Task Force on Sigma Phase is collecting samples of type 316 showing varying degrees of corrosion resistance. A correlation between microstructural characteristics and corrosion resistance will be attempted.

Subcommittee VIII on Specifications for Wrought Products (R. B. Gunia, chairman) prepared the revision of the Specifications for Corrosion-Resisting Steel Bars (A 276 – 55) referred to earlier in the report. A new specification for Cr-Ni-Mn steel bars has been prepared. The emergency alternate provision of Specifications A 314 – 55 have been eliminated and the specification itself has been revised. Both of these actions are being proposed for Society adoption as referred to earlier in this report.

Subcommittee IX on Specifications for Flat Products (G. W. Hinkle, chairman) prepared the revised Specifications A 240, referred to earlier in this report.

A task force was appointed to consider minimum carbon content values for Types 304, 316, 321, 347, and 348. This group is to consult with a similar group in Subcommittee XI.

Subcommittee X on Specifications for Castings (J. J. Kanter, chairman) has approved changing the minimum elongation value and minimum tensile strength value of grade HI and the minimum tensile strength value of grade HK of Specifications A 297. The silicon contents of grades CB-30 and CC-50 of Specifications A 296 were also revised. In addition, the subcommittee has approved the removal of the emergency alternate provisions of Specifications A 296.

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[3] 1955 Book of ASTM Standards, Part 1.

[4] The revised standard appears in the 1958 Book of ASTM Standards, Part 1.

[5] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

These revisions are being balloted through the main committee.

Subcommittee XIII on Specifications for Nuclear Reactor Structural Materials (M. A. Cordovi, chairman) is continuing its task of reviewing present ASTM standards in the light of various proprietary specifications on materials for nuclear plant applications. Although final recommendations from the various task groups are expected by the June meeting, it appears that no new specifications will be required. Recommendations will probably be in the form of additional requirements to existing specifications, these requirements to cover test and inspection methods and maximum limits for various residual elements.

Joint A-1, A-10 Subcommittee on Stainless Tubing Specification (J. J. B. Rutherford, chairman).—Most of the items covered by this subcommittee involved standards under the sole jurisdiction of Committee A-1 and are recorded in the Annual Report of Committee A-1. A number of editorial revisions were made in specifications which are under the joint jurisdiction of Committees A-1 and A-10. However, because of their minor nature, these will not be balloted through Committee A-10 and will be recorded in the report of Committee A-1.

This report has been submitted to letter ballot of the committee. which consists of 103 members; 84 members returned their ballots, of whom 80 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

JEROME STRAUSS,
Chairman.

G. N. EMMANUEL,
Secretary.

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee A-10 presented to the Society through the Administrative Committee on Standards the following recommendations:

Withdrawal of Emergency Alternate Provisions for:

Corrosion-Resisting Chromium-Nickel Steel Plate, Sheet, and Strip (EA - A 167), and Corrosion-Resistant Iron-Chromium and Iron-Chromium-Nickel Alloy Castings for General Application (EA - A 296).

These recommendations were accepted by the Standards Committee on December 3, 1958.

REPORT OF COMMITTEE B-1
ON
WIRES FOR ELECTRICAL CONDUCTORS*

Committee B-1 on Wires for Electrical Conductors held three meetings during the year: in Washington, D. C., on October 25, 1957; in Philadelphia, Pa., on January 21, 1958; and in New York, N. Y., on April 1, 1958. Meetings of Subcommittees II, IV, and VII also were held during the year.

The officers and members-at-large of the Advisory Subcommittee elected for the ensuing term of two years are as follows:

Chairman, D. Halloran.
Vice-Chairman, C. E. Topping.
Secretary, A. A. Jones.
Members-at-large: L. B. Curtis, A. A. Defoe, M. F. W. Heberlein, W. F. Markley, and E. M. Wolf.

The committee accepted with regret the resignation of W. R. Hibbard as Vice-Chairman and wishes to record its appreciation of his many contributions to the committee's work. Mr. Hibbard, although retired, will continue his committee membership on a personal basis.

The committee consists of 85 members, of whom 43 are classified as producers, 27 as consumers, and 15 as general interest members.

NEW TENTATIVES

The committee recommends for publication as tentative the following specifications as appended hereto:[1]

Specification for Half-Hard Aluminum Wire for Electrical Purposes:

Last year the Underwriters' Laboratories expanded the range of tensile strength permitted for intermediate-temper aluminum wire from 17,000 to 22,000 psi, to a new 15,000 to 22,000 psi range. This was done because some users preferred a more ductile wire for smaller size building wires whereas other users preferred the stronger temper for larger cables for duct and conduit installation. Accordingly the new proposed specification is recommended to supplement the present specification for $\frac{3}{4}$-hard aluminum (B 262).

Specification for Rectangular and Square Bare Aluminum Wire for Electrical Conductors:

During the last few years there has been growing commercial interest in rectangular and square aluminum wire for use in magnet wire. About three years ago the committee took note of this trend and started developing suitable specification requirements, as part of its continuing program of developing proposed specifications for several particular types of aluminum conductors.

REVISIONS OF TENTATIVE

The committee recommends revisions as follows of the Tentative Specification for Concentric-Lay-Stranded Aluminum Conductors, Steel Reinforced (ACSR) **(B 232 – 57 T)**[2] and its continuation as tentative:

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* Sixty-first Annual Meeting of the Society, June 22–27, 1958.

[1] The new tentatives appear in the 1958 Book of ASTM Standards, Part 2.

[2] 1957 Supplement to Book of ASTM Standards, Part 2.

Section 12.—Add the following new Paragraph (*b*), relettering the present Paragraph (*b*) as (*c*): "(*b*) There shall be only one length of conductor on a reel."

Table V.—Delete the reference to Footnote *a* from the nominal lengths of conductor sizes No. 5 and No. 6 Awg. Double the nominal lengths of the five constructions where the superscript *a* remains, and double the nominal weights of these constructions. Change footnote *a* to read "[a] See Note 6."

Note 6.—Add to the last sentence "except the random lengths may be not shorter than 25 per cent of the nominal lengths of Table V, which are referenced to footnote *a*."

Adoption of Tentatives as Standard Without Revision

The committee recommends that the following five specifications be approved for reference to letter ballot of the Society for adoption as standard without revision. These specifications have stood for two or three years as tentative, during which time no substantial criticism or suggestions for improvement have been received.

Tentative Specifications for:

Tinned Soft or Annealed Copper Wire for Electrical Purposes (**B 33 - 56 T**),[3]

Lead-Coated and Lead-Alloy-Coated Soft Copper Wire for Electrical Purposes (**B 189 - 56 T**),[3]

Hard-Drawn Aluminum Wire for Electrical Purposes (**B 230 - 55 T**),[4]

Tinned Hard-Drawn and Medium-Hard-Drawn Copper Wire for Electrical Purposes (**B 246 - 56 T**),[3]

Method for Determination of Cross-Sectional Area of Stranded Conductors (**B 263 - 56 T**).[3]

Revisions of Standards Immediate Adoption

The committee recommends for immediate adoption revisions in the following nine standards as indicated, and accordingly asks for the necessary nine-tenths affirmative vote at the Annual Meeting in order that the revisions may be referred to letter ballot of the Society:

Standard Specification for Soft Rectangular and Square Bare Copper Wire for Electrical Conductors (B 48 - 57):[2]

Section 11.—In the heading of the tables in Paragraphs (*b*) and (*c*), change "plus or minus" to read "plus and minus." Change Paragraph (*d*) to read as follows:

> (*d*) The wire shall have rounded corners or rounded edges as specified in Table I and as shown in Fig. 1. Where rounded corners are required the corners of the wire shall be rounded within the limits of radii 25 per cent under and 25 per cent over (as determined by a radius gage) of those values specified in Table I.

Table I.—Change the title and heading to read as follows:

TABLE I.—REQUIREMENTS FOR ROUNDED CORNERS AND ROUNDED EDGES.

Specified Thickness, in.	Specified Width, in.		
	0.751 and Over	0.189 to 0.750, incl	Up to 0.188, incl
	Corner Radius, in.		

Section 14(c).—In the definition of *A*, change "Section 10" to read "Section 12."

Standard Specification for Rope-Lay-Stranded Copper Conductors Having Bunch-Stranded Members, for Electrical Conductors (B 172 - 55):[4]

Section 10(b).—Delete this paragraph.

Standard Specification for Rope-Lay-Stranded Copper Conductors Having Concentric-Stranded Members, for Electrical Conductors (B 173 - 55):[4]

Section 10(b).—Change to read as follows:

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[3] 1956 Supplement to Book of ASTM Standards, Part 2.

[4] 1955 Book of ASTM Standards, Part 2.

(*b*) Where the length of lay is definitely known, the increment may be calculated if desired (Note 9).

Explanatory Note.—Add a new Explanatory Note 9 to read as follows:

NOTE 9.—Any calculation of the increment of weight or electrical resistance, k, of a rope-lay-stranded conductor involves two independent calculations:

1. Determination of the increment due to stranding of the individual members, and
2. Determination of the increment due to twisting these members to form the completed conductor.

In the case of a rope-lay-stranded conductor having concentric-stranded members, the increment k, in per cent, may be expressed as:

$$k = k_m + k_a + \frac{k_m k_a}{100}$$

where:

k_m is the increment of weight or electrical resistance, in per cent, of an individual concentric-stranded member before twisting into the completed conductor, calculated as described in ASTM Specification B 8, Note 9, and

k_a is the increment of weight or electrical resistance, in per cent, due to twisting the concentric-stranded members into the completed conductor, calculated as described in ASTM Specification B 8, Note 9, with each member considered as an individual wire.

Standard Specification for Bunch-Stranded Copper Conductors for Electrical Conductors (B 174 – 55):[4]

Section 10(b).—Delete this paragraph.

Standard Method of Test for Resistivity of Electrical Conductor Materials (B 193 – 57):[2]

Table II.—Add a new group of data, immediately following aluminum, grade EC, to read as follows:

Material	Density, δ, at 20 C, g per cu cm	Temperature Coefficient of Resistance, α, at 20 C
Aluminum Alloy GS10B, per cent IACS:		
57..........	2.70	0.00376
56..........	2.70	0.00370
55..........	2.70	0.00363

Standard Classification of Coppers (B 224 – 52):[4]

Revise as recommended by the Joint Committee on the Classification of Coppers (representing Committees B-1, B-2, and B-5), as set forth in the report of Committee B-5 on Copper and Copper Alloys.

Standard Specification for Concentric-Lay-Stranded Aluminum Conductors, Hard Drawn and Three-Quarter Hard-Drawn (B 231 – 57):[2]

Title.—Revise to read: "Standard Specification for Concentric-Lay-Stranded Aluminum Conductors, Hard-Drawn, Three-Quarter Hard-Drawn, and Half-Hard Drawn (B 231 – 58)."

Section 1(a).—Revise the first sentence to read: "This specification covers concentric-lay-stranded conductors, made of round aluminum wires, either hard-drawn, three-quarter hard-drawn, or half-hard drawn, for general use for electrical purposes."

Section 3(b).—Add at the end of this section, a third reference specification, to read: "Specification for Half-Hard Aluminum Wire for Electrical Purposes (ASTM Designation: B 323)."

Section 7.—Revise the major heading preceding Section 7 to read: "Physical and Electrical Tests on Conductors with Hard-Drawn Aluminum Wires and Conductors Fabricated from Either Three-Quarter Hard-Drawn or Half-Hard Drawn Aluminum Wires and NOT Annealed after Stranding."

In Paragraph (*b*) revise the second, third, and fourth sentences to read as follows:

The minimum breaking strength of hard-drawn, three-quarter hard-drawn, or one-half hard-drawn conductors so tested shall be at least 90 per cent of the total of the calculated minimum breaking strengths of the component wires. The minimum breaking strengths of the component wires shall be calculated using specified nominal wire diameters and specified minimum average tensile strengths in the case of hard-drawn aluminum conductors, or specified minimum tensile strengths in the case of three-quarter hard-drawn and one-half hard-drawn conductors. The maximum breaking strength of conductors made from three-quarter hard-drawn and one-half hard-drawn wires shall be not greater than the sum of the calculated maximum breaking strengths of the component wires.

In Paragraph (*c*) revise the second sentence to read as follows:

> When so tested hard-drawn wires shall have minimum tensile strengths not less than 95 per cent of the tensile strengths prescribed for individual tests in Table I of the Specifications for Hard-Drawn Aluminum Wire for Electrical Purposes (ASTM Designation: B 230), and three-quarter hard-drawn or one-half hard-drawn wires shall have tensile strengths not less than 95 per cent of the minimum tensile strength nor more than 105 per cent of the maximum tensile strength prescribed in Section 5(*a*) of the Specifications for Three-Quarter Hard-Drawn Aluminum Wire for Electrical Purposes (ASTM Designation: B 262) or Section 5(*a*) of the Specification for Half-Hard Aluminum Wire for Electrical Purposes (ASTM Designation: B 323).

Standard Specification for Standard Weight Zinc-Coated (Galvanized) Steel Core Wire for Aluminum Conductors, Steel Reinforced (ACSR) (B 245 – 55):[4]

Table VI.—Correct the fourth from last size (wire diameter) to read "0.1628" instead of "0.8162." Revise the Nominal Coil Weight for sizes 0.1327 and 0.1490 to read "400" instead of "300 lb." Delete the following sizes completely:

Wire Diameter, in.	Type
0.0525	SW
0.0589	SW
0.0835	MW
0.1000	MW
0.0852	MW
0.1186	MW
0.1111	MW
0.1291	MW

Standard Specification for Zinc-Coated (Galvanized) Steel Core Wire (With Coatings Heavier than Standard Weight) for Aluminum Conductors, Steel Reinforced (ACSR) (B 261 – 55):[4]

Table VI.—Revise the Nominal Coil Weight for sizes 0.1327 and 0.1490 to read "400" instead of "300 lb." Delete the following sizes completely:

Wire Diameter, in.	Type
0.0525	SW
0.0589	SW
0.0835	MW
0.1000	MW
0.0852	MW
0.1186	MW
0.1111	MW
0.1291	MW

REAPPROVAL OF STANDARDS

The committee recommends that the following two standards be reaffirmed as standard:

Standard Specifications for:

Medium-Hard-Drawn Copper Wire (**B 2 – 52**), and

Hot-Rolled Copper Rods for Electrical Purposes (**B 49 – 52**).

TENTATIVE CONTINUED WITHOUT REVISION

The committee recommends the continuation without revision of the Tentative Method of Test for Stiffness of Bare Soft Square and Rectangular Copper Wire for Magnet Wire Fabrication (**B 279 – 53 T**).

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[5]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Editorial and Records (A. A. Jones, chairman) continued its editorial review of specifications and coordinated the various subcommittee recommendations regarding revisions of specifications.

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[5] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

Subcommittee II on Methods of Test and Sampling Procedures (R. H. Lloyd, chairman) completed revision of Specification B 193 incorporating representative values of density and temperature coefficient of resistance for aluminum alloy GS10B. It again considered the matter of developing a method of calculating lay factor for bunch-stranded conductors, found no practical mathematical solution, and recommended elimination from Specifications B 172 and B 174 of the provision that where the length of lay is definitely known the increment may be calculated if desired. On request from Committee B-7 on Light Metals and Alloys, a task group is preparing a proposed tentative eddy current method for measuring resistivity.

Subcommittee IV on Conductors of Copper and Copper Alloys (B. J. Sirois, chairman) completed revision of Specification B 48 to clarify the rounded corner and edge requirements, of Specification B 173 to incorporate a method of computing lay factor, and of Specifications B 172 and B 174 to delete provision for computing lay factor (since no practical mathematical solution has been found).

Task groups are cooperating with the U. S. Army Signal Corps Engineering Laboratories in the study of grey-tin transformation, cooperating with Underwriters' Laboratories in an endeavor to coordinate length of lay requirements in small copper conductors, investigating means for measuring wire diameters with a precision greater than 0.1 mil (that is, in hundredths of a mil) and considering degree of significance needed in specifying fine-wire sizes as a preliminary to establishing diameter tolerances for very fine wires, considering a developing need for specifications covering stranded silver-coated conductors having constructions differing from those presently covered in Specification B 286, and cooperating with Committees B-2 and B-5 to survey the possibility of combining in one test method all of the coating test requirements now included in Specifications B 33, B 189, and a proposed Committee B-5 specification for coated strip and sheet.

Subcommittee V on Conductors of Ferrous Metals (L. H. Winkler, chairman) has a task group preparing a proposed specification for aluminum-coated steel core wire for ACSR constructions.

Subcommittee VII on Conductors of Light Metals (A. L. Kolb, chairman) completed preparation of the proposed Tentative Specification for Half-Hard Aluminum Wire for Electrical Purposes and of the proposed Tentative Specification for Rectangular and Square Bare Aluminum Wire for Electrical Conductors. The subcommittee also completed revision of Specification B 231 incorporating one-half hard aluminum strand, and of Specification B 232 limiting all sizes to one length per shipping reel.

Joint Task Groups are considering the preparation of standard test report forms for bare electrical conductors, and a glossary of terms defining the various terms used in specifications under the jurisdiction of Committee B-1.

This report has been submitted to letter ballot of the committee, which consists of 85 members; 55½ members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

D. HALLORAN,
Chairman.

A. A. JONES,
Secretary.

REPORT OF COMMITTEE B-2
ON
NON-FERROUS METALS AND ALLOYS*

Committee B-2 on Non-Ferrous Metals and Alloys met on June 19, 1957, in Atlantic City, N. J. Meetings of the Advisory Committee were held on the same date, and also on December 19, 1957, in New York City.

Mr. George O. Hiers was appointed vice-chairman and secretary of Subcommittee III on White Metal Alloys in line with the policy of the Advisory Committee that vice-chairmen or secretaries be designated for the more active subcommittees.

Mr. Thomas Lippert was appointed chairman and Mr. Arthur Jones secretary of the new Subcommittee IX on Titanium and Titanium Alloys.

Messrs. J. R. Musgrave and A. M. Bounds have been appointed as Committee B-2 representatives to Committee F-1 on Materials for Electron Tubes and Semiconductor Devices.

Messrs. B. J. Sirois and Gordon C. Mutch have been asked to join a subcommittee of Committees B-1 and B-5, which is now considering the Tentative Specification for Lead-Coated and Lead-Alloy-Coated Soft Copper Wire for Electrical Purposes (B 189 - 56 T).

Although Committee B-2 has not conducted any symposia during the past year, nevertheless the year has been an active one, with the revision of specifications under the committee's jurisdiction and the writing of a number of new specifications.

The total membership of Committee B-2 is now 136, of whom 66 are classified as producers, 43 as consumers, and 27 as general interest members.

The officers elected for the ensuing term of two years are as follows:

Chairman, Bruce W. Gonser.
Secretary, A. M. Bounds.

NEW TENTATIVE

The committee recommends for publication as tentative the Specification for Refined Secondary Lead, as appended hereto.[1]

REVISIONS OF TENTATIVES

The committee recommends revisions as indicated of the following tentatives and their continuation as tentative:

Tentative Specifications for Titanium Strip, Sheet, Plate, Bar, Tube, Rod, and Wire (**B 265 - 52 T**):[2] Revise as appended hereto.[1]

Tentative Specifications for Titanium Sponge (**B 299 - 55 T**):[2] Revise as appended hereto.[1]

REVISIONS OF STANDARDS, IMMEDIATE ADOPTION

The committee recommends for immediate adoption revisions as indicated of the following standards and accordingly asks for the necessary nine-tenths affirmative vote at the Annual Meeting in order that the revisions may be referred to letter ballot of the Society:

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* Sixty-first Annual Meeting of the Society, June 22-27, 1958.

[1] The new and revised tentatives appear in the 1958 Book of ASTM Standards, Part 2.

[2] 1955 Book of ASTM Standards, Part 2.

Standard Specifications for Slab Zinc (Spelter) (B 6 – 49):[2]

Table I.—Delete the column headed "Aluminum." Add a new Note 4 to read as follows:

NOTE 4.—Analysis shall not regularly be made for aluminum, but when used for rolled zinc or brass, if found by the purchaser, aluminum shall not exceed 0.005 per cent. Greater amounts may constitute cause for rejection.

Section 9.—In the last line change "galvanized" to read "zinc-coated."

Classification of Coppers (B 224 – 52):[2]
Revise as indicated in the 1958 Report of Committee B-5.[1]

REAPPROVAL OF STANDARD

The committee recommends the reapproval of the Standard Specifications for Rolled Zinc (**B 69 – 39**) which has stood for more than six years without revision.

STANDARDS CONTINUED WITHOUT REVISION

The committee recommends the continuation without revision of the following standards:

Standard Specification for:

- Lake Copper Wire Bars, Cakes, Slabs, Billets, Ingots, and Ingot Bars (**B 4 – 42**),
- Electrolytic Copper Wire, Bars, Cakes, Slabs, Billets, Ingots, and Ingot Bars (**B 5 – 43**),
- White Metal Bearing Alloys (Known Commercially as "Babbitt Metal") (**B 23 – 49**),
- Pig Lead (**B 29 – 55**),
- Nickel (**B 39 – 22**),
- Lead-Coated Copper Sheets (**B 101 – 40**),
- Electrolytic Cathode Copper (**B 115 – 43**),
- Oxygen-Free Electrolytic Copper Wire Bars, Billets, and Cakes (**B 170 – 47**),
- Fire-Refined Copper for Wrought Products and Alloys (**B 216 – 49**), and
- Metallic Antimony (**B 237 – 52**).

TENTATIVES CONTINUED WITHOUT REVISION

The committee recommends the continuation without revision of the Tentative Specification for Fire-Refined Casting Copper (**B 72 – 55 T**).

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[3]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Refined Copper (W. E. Milligan, chairman).—The work which has been done on the classification of coppers as DLP, DHP, or DPA was noted and a task force appointed to determine whether one specification covering all three or three separate new specifications are needed.

It is hoped that Subcommittee I may sponsor a delegation to the International Standards Organization, TC-26, the details of which are being handled by the chairmen of Subcommittees I and II, W. E. Milligan and Sidney Rolle.

Subcommittee II on Refined Lead, Tin, Antimony, and Bismuth (Sidney Rolle, chairman).—The task force which has been handling the preparation of a specification for refined secondary lead has twice balloted on drafts of the specification for this product during the year, finally resulting in agreement as indicated in the above listing of new specifications recommended for adoption. A task group has been appointed to study special lead for nuclear work.

Considerable fact finding has been done in the past few years by working groups under Subcommittee II. These have been primarily concerned with the effect of impurities in tin on the properties of tinplate, electro tinned coatings, and in bronze.

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[3] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

Subcommittee III on White Metal Alloys (Dr. G. H. Clamer, chairman).—The subcommittee's activities during the past year have resulted in the revision of Specifications B 32 and B 284.

Subcommittee IV on Refined Zinc and Wrought Zinc (E. H. Bunce, chairman) is considering a slight revision in Specification B 6 – 49 covering slab zinc. The subcommittee has also indicated a desire that ASTM participate in the proceedings of the International Standards Organization, TC-18, a matter which is now under negotiation.

Subcommittee VII on Refined Nickel and Cobalt, High Nickel Alloys and High Cobalt Alloys, Cast and Wrought (V. J. W. Franceschini, chairman).—This subcommittee has had a most active year during which nearly all the specifications under its jurisdiction have been revised. Specifications B 127, B 160, B 161, B 162, B 163, B 164, B 165, B 166, B 167, and B 168 have all been extensively revised. These cover nickel, nickel-copper alloy, nickel-chromium-iron alloy, nickel-molybdenum, and nickel-molybdenum-chromium alloy in various mill forms.

Subcommittee VIII on Miscellaneous Refined Metals and Alloys (E. E. Schumacher, chairman).—Activity has continued among the nine task groups set up to study the less common metals beryllium, lithium, zirconium and hafnium, columbium, tantalum, thorium, uranium, molybdenum, and tungsten. It has already been indicated that it will not be long before specifications for these once rare metals will be necessary; accordingly the task groups are in the research and fact-gathering stage to prepare for these future requirements. Particular interest has been shown in the Task Group on Zirconium, a metal of great current interest. An active task group is preparing specifications on molybdenum and its alloys.

Subcommittee IX on Titanium and Titanium Alloys (T. W. Lippert, chairman).—After several years of relative inactivity as a task group under Subcommittee VIII, this group has been reorganized as Subcommittee IX and is currently engaged in the revision of Specifications B 265 and B 299, covering titanium strip, sheet, plate, bar, tube, and wire, and titanium sponge.

This report has been submitted to letter ballot of the committee, which consists of 174 members; 81 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

BRUCE W. GONSER,
Chairman.

A. M. BOUNDS,
Secretary.

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee B-2 presented to the Society through the Administrative Committee on Standards the following recommendations:

New Tentative Specification for:

Nickel-Molybdenum and Nickel-Molybdenum-Chromium Alloy Castings (B 332 – 58 T),
Nickel-Molybdenum Alloy Plate and Sheet (B 333 – 58 T),
Nickel-Molybdenum-Chromium Alloy Plate and Sheet (B 334 – 58 T),
Nickel-Molybdenum Alloy Rod (B 335 – 58 T), and
Nickel-Molybdenum-Chromium Alloy Rod (B 336 – 58 T).

Revision of Tentative Specifications for:

Nickel-Copper Alloy Plate, Sheet and Strip (B 127 – 49 T),
Nickel Rod and Bar (B 160 – 49 T),

Nickel Seamless Pipe and Tube (B 161 – 49 T),
Nickel Plate, Sheet, and Strip (B 162 – 49 T),
Seamless Nickel and Nickel Alloy Condenser and Heat Exchanger Tubes (B 163 – 49 T),
Nickel-Copper Alloy Rod and Bar (B 164 – 49 T),
Nickel-Copper Alloy Seamless Pipe and Tube (B 165 – 49 T),
Nickel-Chromium-Iron Alloy Rod and Bar (B 166 – 49 T),
Nickel-Chromium-Iron Alloy Seamless Pipe and Tube (B 167 – 49 T), and
Nickel-Chromium-Iron Alloy Plate, Sheet, and Strip (B 168 – 49 T).

These recommendations were accepted by the Standards Committee on July 25, 1958, and the new and revised tentative specifications appear in the 1958 Book of ASTM Standards, Part 2.

On November 13, 1958, the Standards Committee accepted the following new tentative specifications which are available as separate reprints:

New Tentative Specification for:

Seamless and Welded Unalloyed Titanium Pipe (B 337 – 58 T), and
Seamless and Welded Unalloyed Titanium Tubing (B 338 – 58 T).

REPORT OF COMMITTEE B-3
ON
CORROSION OF NON-FERROUS METALS AND ALLOYS*

Committee B-3 on Corrosion of Non-Ferrous Metals and Alloys held one meeting during the year: at Atlantic City, N. J., on June 19, 1957.

The committee consists of 101 members, of whom 85 are voting members; 34 are classified as producers, 33 as consumers, and 18 as general interest members.

The officers elected for the ensuing term of two years are as follows:

Chairman, K. G. Compton.
Vice-Chairman, E. K. Camp.
Secretary, A. W. Tracy.

TENTATIVES CONTINUED WITHOUT REVISION

The committee recommends the continuation without revision of the following tentatives:

Tentative Methods of:

Salt Spray (Fog) Testing (**B 117 - 57 T**),
Total Immersion Corrosion Test of Non-Ferrous Metals (**B 185 - 43 T**),
Alternate Immersion Corrosion Test of Non-Ferrous Metals (**B 192 - 44 T**), and
Acetic Acid-Salt Spray (Fog) Testing (**B 287 - 57 T**).

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[1]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee III on Spray Test (C. O. Durbin, chairman).—Several members are working on evaluating the effect of copper impurities in the salt solution on results obtained in salt spray tests.

Subcommittee VI on Atmospheric Corrosion (H. R. Copson, chairman) has set November, 1958, as the exposure date for its new atmospheric test program. Twenty-two different alloys of aluminum, some in several heat treatments and some Alclad, a total of 34 groups of specimens, will be exposed at four test locations for 1, 2, 7, and 20 years. There will be a total of 43 alloys of copper, lead, magnesium, molybdenum, nickel, tantalum, titanium, and zinc exposed at the same locations for 2-, 7-, and 20-yr periods. Specimens 8 by 4 by 0.05 in. will be exposed in triplicate for each period at Kure Beach, N. C. (east coast marine), New York Area (Newark, N. J.) (industrial), Point Reyes, Calif. (west coast marine), and State College, Pa. (rural). Corresponding specimens will be stored in duplicate for the same time periods. Amount of corrosion will be determined by weight loss, changes in mechanical properties, and pit depth.

Subcommittee VII on Weather (C. P. Larrabee, chairman) has been studying methods for the determination of sulfur dioxide and chlorides in the atmosphere. The subcommittee, through the Main Committee, sponsored a paper by P. J. Sereda, National Research Council of Canada, on "Progress Report on the

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* Sixty-first Annual Meeting of the Society, June 22–27, 1958.

[1] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

Measurement of Surface Moisture" which was published in the February 1958 ASTM BULLETIN. Messrs. Ellis and Larrabee are drafting a report on the 8-yr exposures of zinc and steel specimens for calibrating the corrosivity of ASTM test sites.

Subcommittee VIII on Galvanic and Electrolytic Corrosion (G. V. Kingsley, chairman) is evaluating the test results of the stainless steel galvanic couples exposed to the atmosphere 15 years at several test sites.

The magnesium panel type of galvanic corrosion test couples are being assembled and will be placed on exposure in the fall of 1958.

This report has been submitted to letter ballot of the committee, which consists of 85 voting members; 69 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

K. G. COMPTON,
Chairman.

A. W. TRACY,
Secretary.

REPORT OF COMMITTEE B-4

ON

METALLIC MATERIALS FOR ELECTRICAL HEATING, ELECTRICAL RESISTANCE AND ELECTRICAL CONTACTS*

Committee B-4 on Metallic Materials for Electrical Heating, Electrical Resistance and Electrical Contacts held three meetings during the year: in Atlantic City, N. J. on June 21, 1957; in Pittsburgh, Pa., on October 18, 1957; and in Washington, D. C., on February 25, 1958.

The committee consists of 51 members, of whom 22 are classified as producers, 21 as consumers, and 8 as general interest members.

As it has done for the past several years, Committee B-4 cooperated with the Pennsylvania State University in sponsoring a Symposium on Electrical Contacts, at the University during the week of June 10, 1957. The same cooperation extends to the symposium which will be held from June 16 to June 18, 1958.

The officers elected for the ensuing term of two years are as follows:

Chairman, Erle I. Shobert II.
Vice-Chairman, Leonard Mayeron.
Secretary, C. K. Strobel.
Assistant Secretary, C. L. Guettel.

A Compilation of ASTM Standards on Thermostatic Bimetals has been issued which contains the standards prepared by Subcommittee III as well as other standards used in bimetal specifications.

NEW TENTATIVE

The committee recommends for publication as tentative the Method of Test for Resistance Characteristics of Microcontacts as appended hereto.[1]

REAPPROVAL OF STANDARDS

The committee recommends the reapproval of the following standards which have stood for more than six years without revision:

Standard Method of Test for:

Resistivity of Metallically Conducting Resistance and Contact Materials (**B 63 - 49**),
Thermoelectric Power of Electrical-Resistance Alloys (**B 77 - 33**),
Linear Expansion of Metals (**B 95 - 39**),
Temperature Resistance Constants of Sheet Materials for Shunts and Precision Resistors (**B 114 - 45**), and
Effect of Controlled Atmospheres Upon Alloys in Electric Furnaces (**B 181 - 50**).

Method for:

Life Test of Electrical Contact Materials (**B 182 - 49**).

Specifications for:

Chromium-Nickel-Iron Alloy Castings (25-12 Class) forHigh-Temperature Service (**B 190 - 50**), and
Nickel-Chromium-Iron Alloy Castings (35-15 Class) for High-Temperature Service (**B 207 - 50**).

Standard Specifications B 83 - 52 and B 267 - 53 T are not recommended for reapproval since they are currently being revised.

The recommendations in this report have been submitted to letter ballot of

the committee, the results of which will be reported at the Annual Meeting.[2]

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* Sixty-first Annual Meeting of the Society, June 22–27, 1958.

[1] The new tentative appears in the 1958 Book of ASTM Standards, Part 3.

ACTIVITIES OF SUBCOMMITTEES

In the Annual Report of 1957, a complete résumé was given of the research activities of the various groups in the committee. This work is continuing and, in general, it is going on as described in that report. Progress has been made in all of the fields of endeavor and the results will be published when new standards have been developed.

In addition, Subcommittee II on Wrought and Cast Alloys for Structural Use at High Temperatures has been reactivated under the chairmanship of W. A. Blonn.

This report has been submitted to letter ballot of the committee, which consists of 51 members; 40 members have returned their ballot, of whom 36 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

ERLE I. SHOBERT II,
Chairman.

C. K. STROBEL,
Secretary.

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[2] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

REPORT OF COMMITTEE B-5

ON

COPPER AND COPPER ALLOYS, CAST AND WROUGHT*

Committee B-5 on Copper and Copper Alloys, Cast and Wrought, held two meetings during the year: at Boston, Mass., in October 1957, and at New York City in January 1958. At these two sessions, the Executive Subcommittee and Subcommittees W-1, W-2, W-3, W-4, F-1, G-1, and G-3 also convened.

The Committee regrets to announce the passing of C. O. Evans, a faithful member of the committee.

The committee wishes to record its regrets in the loss in leadership of G. H. Harnden who has served for over 21 years as a member of Committee B-5. In that time the number of items under the jurisdiction of the committee has more than doubled. Many outstanding accomplishments have been performed during his tenure in office. Mr. Harnden will still continue as a member of the committee.

The committee consists of 134 members, of whom 64 are classified as producers, 49 as consumers, and 19 as general interest.

The officers elected for the ensuing term of two years are as follows:

Chairman, W. H. Jennings.
First Vice-Chairman, W. D. France.
Second Vice-Chairman, W. Romanoff.
Secretary, L. H. Adam.

The Supervisory Group to the American Standard Assn. on International Organization for Standardization, Technical Committee ISO/TC-26 on Copper and Copper Alloys, held two meetings: at Boston, Mass., in October 1957, and at New York, N. Y., in January 1958.

Three working groups have been established on current activities pertaining to ISO/TC-26: Mechanical Testing, B. L. Wilson, chairman; Classification of Wrought Copper and Copper Alloys, A. E. Moredock, chairman; and Classification of Cast Copper and Copper Alloys, J. S. Roberts, chairman.

Items resulting from the meeting held in Stockholm, Sweden, June 13 and 14, 1955, pertaining to the expansion test, mercurous nitrate test, and classification of coppers were submitted by letter ballot and recommendations made to the Secretariat of ASA to cast an affirmative ballot on the changes.

A forthcoming meeting of the group is to take place at Harrogate, England, with representatives of the United States in attendance. Sidney Rolle has gone to considerable effort in soliciting support for this activity. Current plans for the meeting will consist of continuation of the work on classification of cast copper alloys, standardization of the expansion test and mercurous nitrate test, and classification of coppers. The ASTM Specifications for Electrolytic Copper Wire Bars, Cakes, Slabs, Billets, Ingots, and Ingot Bars (B 5 – 43) is to be proposed for international standardization. Other items may arise at the request of foreign delegates.

Two excellent technical sessions were

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* Sixty-first Annual Meeting of the Society, June 22–27, 1958.

held by Committee B-5, with the preentation of papers entitled "Nuclear Radiation Effect on Copper and Copper Alloys" by John Moteff, Technical Design, General Electrical Co., and "The Mechanical and Physical Characteristics of Jewelry Bronze, 87.5 per cent Strip" by Delmar E. Trout, Research Metallurgist, Scovill Manufacturing Co. The first paper is appended hereto;[1] the second was published in the ASTM BULLETIN, No. 227, Jan. 1958.

RECOMMENDATIONS AFFECTING STANDARDS

The committee is recommending the revision of 35 standards and 8 tentatives. These recommendations are covered in detail in the Appendix.[2]

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[3]

ACTIVITIES OF SUBCOMMITTEES

The Executive Subcommittee held meetings prior to each main committee meeting. The committee is sponsoring a forthcoming Copper and Brass Industry luncheon to be held at Boston, June, 1958.

A proposed revision of Classification of Copper (B 224) was submitted to letter ballot of the committee, with no negative ballots cast. The revised classification is appended hereto.[4]

A joint task group to review methods of test of tin and lead alloy coatings was established in conjunction with Committee B-1 on Wires for Electrical Conductors, B. J. Sirois, chairman.

An item pertaining to the request for closer tolerances on strip standards was submitted to the board of directors for study.

Subcommittee W-1 on Plate, Sheet and Strip (A. E. Moredock, chairman) held two meetings and took action on the following items: A revision of Specification B 103 submitted to letter ballot on tin and phosphorus limits for improved fatigue properties was referred back to the subcommittee for resolution of negative ballots. Revisions of Specification B 194 to include a new hardness conversion table based on task group study on thin strip is being referred to letter ballot of the subcommittee. Editorial revisions on resistivity terminology were recommended for Specification B 152. It was recommended that editorial revisions be made with respect to reference to Methods E 8 in Specifications B 248, B 11, B 19, B 96, B 100, B 130, B 171, and B 291. Specification B 291 is being recommended for adoption as standard.

Subcommittee W-2 on Rods, Bars, and Shapes (J. D. MacQueen, chairman) held two meetings and discussed editorial changes to appear in the following specifications: B 249, B 12, B 187, B 283, B 301, B 16, B 21, B 98, B 133, B 138, B 139, B 140, B 150, and B 151. Revisions of Specification B 140 are approved and appear in the Appendix to this report.[2]

Subcommittee W-3 on Wire and Wire Rod (Sidney Metzger, chairman) held two meetings and recommended revisions of Specifications B 159 and B 250 as indicated in the Appendix. The Subcommittee also recommended the reapproval of Specifications B 134 and B 197 as standard, the adoption of Specification B 250 as standard, and the continuation of Specification B 272 as tentative.

Subcommittee W-4 on Pipe and Tube

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[1] See p. 159.

[2] See p. 144.

[3] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

[4] The revised classification appears in the 1958 Book of ASTM Standards, Part 2.

(G. C. Mutch, chairman).—Action was taken on the revision of the following specifications to include references to details on methods of tests: B 13, B 42, B 43, B 68, B 75, B 88, B 111, B 135, B 188, B 251, B 280, B 302, B 306, and B 315.

Subcommittee F-1 on Castings, and Ingots for Remelting (G. H. Clamer, chairman) held two meetings and, based on the investigative work of a task group headed by Mr. Romanoff, Specification B 30 is being modified to include new editorial format and with specific technical requirements. Revisions in test bar dimensions in Specifications B 198 and Recommended Practice B 208 were made to eliminate discrepancies. A discussion was held on the method of compression testing in Standard B 22. The subcommittee recommended reapproval of the following specifications: B 52, B 53, B 61, B 62, B 132, B 143, B 144, B 145, B 146, B 147, B 148, and B 149. Four specifications which are currently being investigated, B 22, B 66, B 67, and B 119, were not recommended for reapproval but are being retained in their present status.

Subcommittee G-1 on Methods of Test (G. R. Gohn, chairman).—As a result of activity in ISO/TC-26, studies were made on Specifications B 153 and B 154 resulting in modifications which were approved by the committee.

Subcommittee G-2 on Tolerances (J. E. McGraw, chairman) held no meetings but have under consideration a number of tolerance modifications requested by members of industry.

Subcommittee G-3 on Editorial and Publications (W. F. Roeser, chairman) held two meetings and is diligently studying the application of the proposed recommendation of form of ASTM Specifications to Committee B-5 product specifications.

This report has been submitted to letter ballot of the committee, which consists of 134 voting members; 83 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

G. H. HARNDEN,
Chairman.

L. H. ADAM,
Secretary.

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee B-5 presented to the Society through the Administrative Committee on Standards the following recommendations:

Revision of Tentative Specifications for:

General Requirements for Wrought Copper and Copper-Alloy Wire (B 250 – 58 T), and Rectangular Copper Wire for General Purposes (B 272 – 52 T).

These recommendations were accepted by the Standards Committee on September 9, 1958, and the revised tentative specifications are available as separate reprints.

APPENDIX I

RECOMMENDATIONS AFFECTING STANDARDS FOR COPPER AND COPPER ALLOYS, CAST AND WROUGHT

In this Appendix are given the recommendations affecting certain standards covering copper and copper alloys which are referred to earlier in this report. The standards appear in their present form in the 1955 Book of ASTM Standards, Part 2, or in the 1956 or 1957 Supplements to Book of ASTM Standards, Part 2.

REVISIONS OF TENTATIVES

The committee recommends revisions as indicated of the following tentatives and their continuation as tentative:

Tentative Specification for General Requirements for Wrought Copper and Copper-Alloy Plate, Sheet, Strip, and Rolled Bar (B 248 – 55 T):

Section 5.—Add to the table a Footnote *b*, referenced to Methods E 8, to read as follows: "[b] The tension test specimen shall conform to the dimensions prescribed in Sections 6, 7, 8, or 9 of Methods E 8 – 58 T."

Tentative Specification for General Requirements for Wrought Copper and Copper-Alloy Rod, Bar, and Shapes (B 249 – 55 T):

Section 5.—Add to the table a Footnote *b*, referenced to Method E 8, to read to read "[b] The tension test specimen shall conform to the dimensions prescribed in Sections 8, 10, 11, or 12 of Methods E 8 – 58 T."

Add a new sentence at the end of the section to read "Whenever tension test results are obtained from both full size and from machined test specimens and they differ, the results obtained from full size test specimens shall be used to determine conformance to the specification requirements."

Tentative Specification for General Requirements for Wrought Copper and Copper-Alloy Wire (B 250 – 55 T):

Tables V, VII, VIII, IX, and X.—In each table add a reference to ASTM Designation B 197 to the line in parentheses immediately following the table title.

Tentative Specification for General Requirements for Wrought Seamless Copper and Copper-Alloy Pipe and Tube (B 251 – 56 T):

Section 2(c).—Revise to read "(*c*) *Average Diameter.*—The average outside or inside diameter of a tube is the average of the maximum and minimum outside diameters or the maximum and minimum inside diameters, whichever is applicable, as determined at any one cross-section of the tube."

Section 4(c).—Change the words "physical tests" wherever they appear to "mechanical tests."

Section 5.—In Paragraph (*a*), in the tabulation of tests add the following in parentheses to the line "Tension . . . E 8": "(See also 5(*b*), (*c*), and (*d*) ff.)"

Add new Paragraphs (*b*), (*c*) and (*d*) to read as follows:

(*b*) Tension test specimens shall be of the full section of the tube and shall conform to the requirements of Section 13(*a*) of Methods E 8, unless the limitations of the testing machine preclude the use of such a specimen. Test specimens conforming to type No. 1 of Fig. 13 of Methods E 8 may be used when a full section specimen cannot be tested.

(*c*) Whenever tension test results are obtained from both full size and from machined test specimens and they differ, the results obtained from full size test specimens shall be used to determine conformance to the specification requirements.

(*d*) Tension test results on material covered by this specification are not seriously affected by variations in speed of testing. A considerable range of testing speed is permissible; however, the rate of stressing to the yield strength should not exceed 100,000 psi per minute. Above the yield strength the movement per minute of the testing machine head under load should not exceed 0.5 in. per in. of gage length (or distance between grips for full section specimens).

Reletter the present Paragraph (*b*) as (*e*) and revise to read "(*e*) The surface of the test specimen for microscopic examination shall approximate a radial longitudinal section of the tube."

Section 8.—Revise Paragraph (*f*) to read:

(*f*) *Roundness* (Applicable to ASTM Designations B 42, B 43, B 75, B 88, and B 135). For drawn unannealed tube or pipe in straight lengths, the roundness tolerances shall be as follows:

t/D (Ratio of Nominal Wall Thickness to Nominal Outside Diameter)	Roundness Tolerance as Per Cent of Nominal Outside Diameter (Expressed to the Nearest 0.001 in.)
0.01 to 0.03 incl.	1.5
Over 0.03 to 0.05 incl.	1.0
Over 0.05 to 0.10 incl.	0.8 or 0.002 in., whichever is greater
Over 0.10	0.7 or 0.002 in., whichever is greater

Compliance with the roundness tolerance shall be determined by taking measurements on the outside diameter only, irrespective of the manner in which the tube dimensions are specified.

The deviation from roundness is measured as the difference between major and minor diameters as determined at any one cross-section of the tube.

No tolerances have been established for as-extruded tube, redraw tube, annealed tube, any tube furnished in coils or drawn tube whose wall thickness is under 0.016 in.

Change the second sentence of Paragraph (*g*) to read "The standard length for copper water tube furnished in straight lengths shall be 20 ft."

Change the first reference to B 88 in Paragraph (*h*) (*5*) to read "For straight lengths of 20 ft. the tolerance shall be all plus 1 in."

Delete Paragraph (*h*) (*3*) and renumber subsequent paragraphs accordingly.

Table VIII.—Delete references to B 68 and B 75.

Tentative Specification for Seamless Tube for Refrigeration Field Service (B 280 – 55 T):

Section 7.—Change the words "tensile properties" to read "mechanical requirements."

Table II.—Add two new sizes, $\frac{3}{4}$ in. and $\frac{7}{8}$ in., as follows:

Nominal Size, in.		Internal Area per Length of Tube, sq ft		Residue Limit, g per 50-ft coil
Outside Diameter	Wall Thickness	1 ft	50 ft	
¾	0.042	0.1744	10.46[a]	0.0366[a]
⅞	0.045	0.2055	12.33[a]	0.0432[a]

[a] 60-ft coil (see Section 17 (*b*)).

Section 14.—Change the present Section 14 to 14(*a*). In the tabulation of tests, revise one line as follows:

Tension . . . E 8 (see also Sections 14(*b*), (*c*), and (*d*) ff.)

Add new Paragraphs (*b*), (*c*), and (*d*) as follows:

(*b*) Tension test specimens shall be of the full section of the tube and shall conform to the requirements of Section 13(*a*) of ASTM Methods E 8, unless the limitations of the testing machine preclude the use of such a specimen. Test specimens conforming to type No. 1 of Fig. 13 (Section 13(*b*)) of ASTM Methods E 8 may be used when a full section specimen cannot be tested.

(*c*) Whenever tension test results are obtained from both full size and from machined test specimens and they differ, the results obtained from full size test specimens shall be used to determine conformance to the specification requirements.

(*d*) Tension test results on material covered by this specification are not seriously affected by variations in speed of testing. A considerable range of testing speed is permissible; however, the rate of stressing to the yield strength should not exceed 100,000 psi per minute. Above the yield strength the movement per minute of the testing machine head under load should not exceed 0.5 in. per in. of gage length (or distance between grips for full section specimens).

Table III.—Add two new sizes, ¾ in. and ⅞ in., as follows:

Nominal Size, in.	Actual Outside Diameter, in.	Wall Thickness, in.	Weight, lb per ft	Average Outside Diameter, plus and minus, in.	Wall Thickness, plus and minus, in.
¾....	0.750	0.042	0.362	0.0025	0.0035
⅞....	0.875	0.045	0.455	0.003	0.004

Section 17(b).—Revise to read as follows: "The standard length for coils shall be 50 ft except for the nominal sizes ¾ in. and ⅞ in., which shall be 60 ft. The length tolerances for all coils shall be plus 12 in. and minus 0 in."

Tentative Specification for Copper-Zinc-Manganese Alloy (Manganese Brass) Sheet and Strip (B 291 – 55 T):

Section 11.—Add a footnote (*b*) referenced to Methods E 8 to read "The tension test specimen shall conform to the dimensions shown in Fig. 7 of Methods E 8 – 58 T."

Tentative Specification for Tellurium Copper Rod (B 301 – 55 T):

Section 7.—Add a Note to the second sentence to read "NOTE.—The tension test specimens shall conform to the dimensions prescribed in Sections 8, 10, 11, or 12 of Methods E 8 – 58 T."

Tentative Specification for Copper-Silicon Alloy Seamless Pipe and Tube (B 315 – 57 T):

Section 2(c).—Revise to read as follows:

> The average outside or inside diameter of a tube is the average of the maximum and minimum outside diameters or the maximum and minimum inside diameters, whichever is applicable, as determined at any one cross-section of the tube.

Section 7.—Change the words "Tensile properties" where they appear to "tensile requirements."

Section 8(b).—Revise the second sentence to read "The pipe or tube need not be tested at a hydrostatic pressure of over 1000 psi unless so specified."

Section 9(c).—Change the words "physical tests" to "mechanical tests."

Section 10.—Change the present Section 10 to 10(*a*). In the tabulation of tests, revise one line to read as follows:

> Tension . . . E 8 (See also Sections 10(*b*), (*c*), and (*d*) ff.)

Add new Paragraphs (*b*), (*c*), and (*d*) to read as follows:

> (*b*) Tension test specimens shall be of the full section of the tube and shall conform to the requirements of Section 13(*a*), ASTM Methods E 8, unless the limitations of the testing machine preclude the use of such a specimen. Test specimens conforming to type No. 1 of Fig. 13 (Section 13(*b*) of ASTM Methods E 8) may be used when a full section specimen cannot be tested.
>
> (*c*) Whenever tension test results are obtained from both full size and from machined test specimens and they differ, the results obtained from full size test specimens shall be used to determine conformance to the specification requirements.

(*d*) Tension test results on material covered by this specification are not seriously affected by variations in speed of testing. A considerable range of testing speed is permissible; however, the rate of stressing to the yield strength should not exceed 100,000 psi per minute. Above the yield strength the movement per minute of the testing machine head under load should not exceed 0.5 in. per in. of gage length (or distance between grips for full section specimens).

REVISIONS OF STANDARDS, IMMEDIATE ADOPTION

The committee recommends for immediate adoption revisions as indicated of the following standards, and accordingly asks for the necessary nine-tenths affirmative vote at the Annual Meeting in order that the revisions may be referred to letter ballot of the Society:

Standard Specification for Copper Plates for Locomotive Fireboxes (B 11 - 55):

Section 8.—In line 8 refer to Fig. 6 of Methods E 8 - 58 T instead of the present Fig. 1. Delete the present Fig. 1 and its accompanying Notes 1, 2, and 3.

Standard Specification for Copper Rods for Locomotive Staybolts (B 12 - 55):

Section 8.—Add a Note to Section 8 to read "NOTE.—The full size section of the tension test specimen may be slightly reduced as prescribed in Section 10(*a*) (*1*) of Methods E 8 - 58 T to insure fracture within the gage length."

Standard Specification for Seamless Copper Boiler Tubes (B 13 - 57):

Section 6.—Add three new paragraphs to the Note at the end of the section to read as follows:

> Tension test specimens shall be of the full section of the tube and shall conform to the requirements of Section 13(*a*) of ASTM Methods E 8, unless the limitations of the testing machine preclude the use of such a specimen. Test specimens conforming to type No. 1 of Fig. 13 (Section 13(*b*)) of ASTM Methods E 8 may be used when a full section specimen cannot be tested.
>
> Whenever tension test results are obtained from both full size and from machined test specimens and they differ, the results obtained from full size test specimens shall be used to determine conformance to the specification requirements.
>
> Tension test results on material covered by this specification are not seriously affected by variations in speed of testing. A considerable range of testing speed is permissible; however, the range of stressing to the yield strength should not exceed 100,000 psi per minute. Above the yield strength the movement per minute of the testing machine head under load should not exceed 0.5 in. per in. of gage length (or distance between grips for full section specimens).

Standard Specification for Free-Cutting Brass Rod, Bar, and Shapes for Use in Screw Machines (B 16 - 52):

Section 6.—Add a Note to the second sentence to read "NOTE.—The tension test specimens shall conform to the dimensions prescribed in Section 8, 10, 11, or 12 of Methods E 8 - 58 T."

Add a new sentence reading "Whenever tension test results are obtained from both full size and from machined test specimens and they differ, the results obtained from full size test specimens shall be used to determine conformance to the specification requirements."

Standard Specification for Cartridge Brass Sheet, Strip, Plate, Bar, and Disks (B 19 - 55):

Section 13(a).—Change Footnote *c* under the tabulation of methods to read "[c] The tension test specimen shall conform to the dimensions shown in Figs. 7 or 8 of Methods E 8 - 58 T."

Standard Specification for Naval Brass Rod, Bar, and Shapes (B 21 - 54):

Section 7(a).—Following the second sentence add a Note to read "NOTE.—The tension test specimens shall conform to the dimensions prescribed in Sections 8, 10, 11, or 12 of Methods E 8 - 58 T."

TABLE I.—COIL LENGTH TOLERANCES FOR SPECIFIC LENGTHS, ALL PLUS (IN IN.).

(New Table I, Specification B 68 – 55)

Outside Diameter, in.	For Nominal Lengths (in ft)	
	Up to 50 incl	Over 50 to 100 incl
Up to 2 incl.	12	24

TABLE II.—COIL LENGTH TOLERANCES FOR MILL LENGTHS, ALL PLUS.

(In per cent of nominal length)

(New Table II, Specification B 68 – 55)

Outside Diameter, in.	Applicable Only to Full Length Pieces	
	For lengths up to 100 ft incl, per cent	For lengths over 100 ft to 2000 ft. incl, per cent
Up to 1 incl.	5[a] or 2 ft, whichever is greater	10[a]
Over 1 to 2 incl.	5[a] or 2 ft, whichever is greater	No tolerances established

[a] Expressed to the nearest foot.

Add a new sentence reading "Whenever tension test results are obtained from both full size and from machined test specimens and they differ, the results obtained from full size test specimens shall be used to determine conformance to the specification requirements."

Standard Specification for Seamless Copper Pipe, Standard Sizes (B 42 – 57):

Section 9(b).—Revise the second sentence to read "The pipe need not be tested at a hydrostatic pressure of over 1000 psi unless so specified."

Standard Specification for Seamless Red Brass Pipe, Standard Sizes (B 43 – 57):

Section 9(b).—Revise the second sentence to read "The pipe need not be tested at a hydrostatic pressure of over 1000 psi unless so specified."

TABLE III.—SCHEDULE OF MILL LENGTHS WITH ENDS FURNISHED IN COILS.

(New Table III, Specification B 68 – 55)

Outside Diameter, in.	Nominal Length, ft	Shortest Permissible Length, per cent of nominal length	Maximum Permissible Weights of Ends, in per cent of Lot Weight
Up to 1 incl.	Up to 100 incl	70[a]	10
Over 1 to 2 incl.	Up to 100 incl	60[a]	20
Up to 1 incl.	Over 100 to 2000 incl	50	50[b]

[a] Expressed to nearest foot.

[b] Short pieces may be included as follows: up to 10 per cent of lot weight between 50 ft and one quarter of full length; and up to 40 per cent between one quarter and full length.

Standard Specification for Seamless Copper Tube, Bright Annealed (B 68 – 55):

Section 5(b).—Delete this section.

Section 6.—Change the words "tensile properties" to read "mechanical requirements."

Section 10(d).—Revise to read "*Length Tolerances.*—Length tolerances shall be as given in Tables I, II, and III."

New Tables.—Add the accompanying new Tables I, II, and III.

Standard Specification for Seamless Copper Tube (B 75 – 57):

Section 3.—Add a new item (*3*) to read as follows, renumbering subsequent items accordingly: "(*3*) Whether tension tests are required (for drawn tempers only) (see Section 7)."

Section 5.—Delete this section and replace with new Sections 5 and 6, to read as follows, renumbering subsequent sections accordingly:

5. *Tempers of Drawn Tube.*—The tempers of drawn tube shall be designated as light-drawn, drawn, and hard-drawn (Table II). Light-drawn (bending) temper is used only when a tube of some stiffness but yet capable of being bent is needed. Drawn temper is for general purposes and is most commonly used where there is no specific requirement for high strength on the one hand, or for bending qualities on the other. Hard-drawn temper is used only where there is need for a tube as strong as is commercially feasible for the sizes indicated. For any combination of diameter and wall thickness not covered under hard-drawn temper, the values given for drawn temper shall be used.

TABLE IV.—MECHANICAL PROPERTY REQUIREMENTS OF DRAWN TEMPER TUBE.

(Revision of Table II, Specification B 75 – 57)

Drawn Temper	Outside Diameter, in.	Wall Thickness, in.	Rockwell Hardness[a]		Tensile Strength, psi
			Scale	Hardness	
Light	all	all	30T	30 to 60	36 000 to 47 000
Drawn (General Purpose)	all	all	30T	30 min	36 000 min
Hard	up to 1	0.020 to 0.120 incl	30T	55 min	45 000 min
	over 1 to 2 incl	0.035 to 0.180 incl			
	over 2 to 4 incl	0.060 to 0.250 incl			

[a] Rockwell hardness values shall apply only to tubes having an inside diameter of $\frac{5}{16}$ in. or over and a wall thickness of 0.020 in. and over. Rockwell hardness tests shall be made on the inside surface of the tube. When suitable equipment is not available for determining the specified Rockwell hardness, other Rockwell scales and values may be specified subject to agreement between purchaser and supplier.

TABLE V.—MECHANICAL PROPERTY REQUIREMENTS OF ANNEALED TEMPER TUBE.

(New Table III, Specification B 75 – 57)

Annealed Temper	Outside Diameter, in.	Wall Thickness, in.	Rockwell Hardness[a]		Average Grain Size, mm
			Scale	Value	
Soft	all	0.015 to 0.035	15T	60 max	0.040 min
		0.035 and over	F	50 max	0.040 min
Light	all	0.015 to 0.035	15T	65 max	0.040 max
		0.035 and over	F	55 max	0.040 max

[a] Rockwell hardness values shall apply only to tube having an inside diameter of $\frac{5}{16}$ in. or over and having a wall thickness of 0.015 in. or over. For all other tube no Rockwell values shall apply. Rockwell hardness tests shall be made on the inside surface of the tube. When suitable equipment is not available for determining the specified Rockwell hardness, other Rockwell scales and values may be specified subject to agreement between purchaser and supplier.

6. *Tempers of Annealed Tube.*—The tempers of annealed tube shall be designated as light anneal and soft anneal (Table III).

Section 6.—Change the title to read "Mechanical Properties of Drawn Temper Tube." Renumber as Section 7, and revise to read as follows:

7. Drawn temper tube shall conform to the mechanical properties prescribed in Table II. Tension tests are required for tubes under $\frac{5}{16}$ in. inside diameter or with a wall thickness under 0.020 in. The tension test for other sizes of tube need not be made except when indicated by the purchaser at the time of placing the order. A convenient method of indicating that the tension test is required is to state that "Test procedure 'T' is required." (See Section 3 (*3*).) Where agreement on the Rockwell hardness tests cannot be reached, the tensile strength requirements of Table II shall be the basis for acceptance or rejection.

Table II.—Revise Table II to read as shown in the accompanying Table IV.

TABLE VI.—COIL LENGTH TOLERANCES, SPECIFIC LENGTHS, ALL PLUS (IN IN.).

(New Table IV, Specification B 75 – 57)

Outside Diameter, in.	For Nominal Lengths (in ft)	
	Up to 50 incl	Over 50 to 100 incl
Up to 2 incl.	12	24

TABLE VII.—COIL LENGTH TOLERANCES, MILL LENGTHS, ALL PLUS.

(In per cent of nominal length)

(New Table V, Specification B 75 – 57)

Outside Diameter, in.	Applicable Only to Full Length Pieces	
	For lengths up to 100 ft incl, per cent	For lengths over 100 to 2000 ft incl, per cent
Up to 1 incl. . .	5[a] or 2 ft, whichever is greater	10[a]
Over 1 to 2 incl.	5[a] or 2 ft, whichever is greater	no tolerances established

[a] Expressed to the nearest foot.

New Section.—Add a new Section 8 to read as follows:

> 8. *Mechanical Property Requirements of Annealed Temper Tube.*—Annealed temper tube shall conform to the grain size and Rockwell hardness limits prescribed in Table III.

New Table.—Add a new Table III to read as shown in the accompanying Table V.

Note.—Transfer the Note following the present Section 6 to follow the new Section 8.

Section 8.—Renumber the present Section 8 as Section 10. Revise Paragraph (*b*) to read "The tube need not be tested at a hydrostatic pressure of over 1000 psi unless so specified."

Section 12(d).—Revise to read "(*d*) *Length Tolerances.*—Length tolerances shall be as given in Tables IV, V, and VI."

New Tables.—Add new Tables IV, V, and VI as shown in the accompanying Tables VI, VII, and VIII.

TABLE VIII.—SCHEDULE OF MILL LENGTHS WITH ENDS FURNISHED IN COILS.

(New Table VI, Specification B 75 – 57)

Outside Diameter, in.	Nominal Length, ft	Shortest Permissible Length, ft, per cent of nominal length	Maximum Permissible Weight of Ends, in per cent of Lot Weight
Up to 1 incl. . .	Up to 100 incl	70[a]	10
Over 1 to 2 incl.	Up to 100 incl	60[a]	20
Up to 1 incl. . .	Over 100 to 2000 incl	50	50[b]

[a] Expressed to nearest foot.

[b] Short pieces may be included as follows: up to 10 per cent of lot weight between 50 ft and one-quarter of full length; and up to 40 per cent between one-quarter and full length.

Standard Specification for Seamless Copper Water Tube (B 88 – 55):

Section 3.—Add a new item (*3*) to read as follows, renumbering subsequent items accordingly: "Whether tension tests and grain size determinations are required (Section 8)."

Section 8.—Change the title to "Mechanical Properties" and revise to read as follows:

> 8. The tube shall conform to the mechanical property requirements prescribed in Table I. Tension tests and grain size determinations need not be made except when indicated by the purchaser at the time of placing the order. A convenient method of indicating that these tests are to be made is to state that "Test Procedure 'T' is required" (Section 3 (*3*)). Where agreement on the Rockwell hardness tests cannot be reached, the tensile strength and grain size requirements of Table I shall be the basis for acceptance or rejection.

Table I.—Revise to read as shown in the accompanying Table IX.

Section 10(b).—Revise the second sentence to read "The tube need not be tested at a hydrostatic pressure of over 1000 psi unless so specified."

Standard Specification for Copper-Silicon Alloy Plate and Sheet for Pressure Vessels (B 96 – 55):

Section 7.—Change Footnote *b* under the tabulation of methods to read “[b] The tension test specimen shall conform to the dimensions shown in Figs. 7 or 8 of Methods E 8 – 58 T.”

Standard Specification for Copper-Silicon Alloy Rod, Bar, and Shapes (B 98 – 55):

Section 6.—Add a Note following the second sentence to read “Note.—The tension test specimens shall conform to the dimensions prescribed in Sections 8, 10, 11, or 12 of Methods E 8.”

Add a new sentence to read “Whenever tension test results are obtained from both full size and from machined test specimens and they differ, the results obtained from full size test specimens shall be used to determine conformance to the specification requirements.”

Standard Specification for Rolled Copper-Alloy Bearing and Expansion Plates and Sheets for Bridge and Other Structural Uses (B 100 – 55):

Section 7(a).—Change Footnote *b* under the tabulation of methods to read “[b] The tension test specimen shall conform to the dimensions shown in Figs. 7 or 8 of Methods E 8 – 58 T.”

TABLE IX.—MECHANICAL PROPERTY REQUIREMENTS.
(Revision of Table I, Specification B 88 – 55)

Temper	Nominal Size	Rockwell Hardness[a]		Tensile Strength, min, psi	Average Grain Size, mm
		Scale	Value		
Annealed, coils	all	F	50 max	30 000	0.040 min
Annealed, straight lengths	all	F	55 max	30 000	0.025 min
Drawn	all	30T	30 min	36 000	...

[a] Rockwell hardness tests shall be made on the inside surfaces of the tube. When suitable equipment is not available for determining the specified Rockwell hardness, other Rockwell scales and values may be specified subject to agreement between purchaser and supplier.

Standard Specification for Copper and Copper-Alloy Seamless Condenser Tubes and Ferrule Stock (B 111 – 57):

Section 7(b).—Delete this paragraph.

Section 11.—Revise the second sentence to read “The tube need not be tested at a hydrostatic pressure of over 1000 psi unless so specified.”

Section 12.—Renumber this paragraph as 12(*a*).

Add to the tabulation of tests the following line:

Tension . . . E 8.

Add a new Paragraph (*b*) to read as follows:

The surface of the test specimen for microscopic examination shall approximate a radial longitudinal section of the tube.

Table II.—Add three new paragraphs to Footnote *a* to read as follows:

Tension test specimens shall be of the full section of the tube and shall conform to the requirements of Section 13(*a*) of ASTM Methods E 8, unless the limitations of the testing machine preclude the use of such a specimen. Test specimens conforming to type No. 1 of Fig. 13 (Section 13(*b*)) of ASTM Methods E 8 may be used when a full section specimen cannot be tested.

Whenever tension test results are obtained from both full size and from machined test specimens and they differ, the results obtained from full size test specimens shall be used to determine conformance to the specification requirements.

Tension test results on material covered by this specification are not seriously affected by variations in speed of testing. A considerable range of testing speed is permissible; however, the range of stressing to the yield strength should not exceed 100,000 psi per minute. Above the yield strength the movement per minute of the testing machine head under load should not exceed 0.5 in. per in. of gage length (or distance between grips for full section specimens).

Add a third sentence to read "Whenever tension test results are obtained from both full size and from machined test specimens and they differ, the results obtained from full size test specimens shall be used to determine conformance to the specification requirements."

TABLE X.—MECHANICAL PROPERTY REQUIREMENTS OF DRAWN TEMPER TUBE.
(Revision of Table II, Specification B 135 – 55)

Alloy No.	Temper	Outside Diameter, in	Wall Thickness, in.	Tensile Strength, psi	Rockwell Hardness,[a] 30T
1	Light-drawn	all	all	44 000 to 58 000	43 to 75
	Drawn (general purpose)	all	all	44 000 min	43 min
	Hard-drawn	up to 1 incl	0.020 to 0.120 incl	57 000 min	65 min
		over 1 to 2 incl	0.035 to 0.180 incl		
		over 2 to 4 incl	0.060 to 0.250 incl		
2, 3 and 4	Drawn (general purpose	all	all	54 000 min	53 min
	Hard-drawn	up to 1 incl	0.020 to 0.120 incl	66 000 min	70 min
		over 1 to 2 incl	0.035 to 0.180 incl		
		over 2 to 4 incl	0.060 to 0.250 incl		
5 and 6	Drawn (general purpose)	all	all	54 000 min	55 min

[a] Rockwell hardness values shall apply only to tubes having an inside diameter of $\frac{5}{16}$ in. or over and a wall thickness of 0.012 in. and over. Rockwell hardness shall be made on the inside surface of the tube. When suitable equipment is not available for determining the specified Rockwell hardness, other Rockwell scales and values may be specified subject to agreement between purchaser and supplier.

Standard Specification for Commercial Bronze Strip (B 130 – 55):

Section 13(a).—Change Footnote *c* under the tabulation of methods to read "[c] The tension test specimen shall conform to the dimensions shown in Fig. 7 of Methods E 8 – 58 T."

Standard Specification for Copper Rod, Bar and Shapes (B 133 – 57):

Section 12(a).—Add a note to the second sentence to read "NOTE.—The tension test specimens shall conform to the dimensions prescribed in Sections 8, 10, 11, or 12 of Methods E 8 – 58 T."

Standard Specification for Seamless Brass Tube (B 135 – 55):

Section 3.—Add a new item (*3*) to read as follows, renumbering subsequent items accordingly: "(*3*) Whether tension tests are required (for drawn tempers only) (Section 7)."

Section 5.—Revise to read as follows:

5. The tempers of drawn tube shall be designated as light-drawn, drawn, and hard-drawn (see Table II). Light-drawn (bending) temper is used only when a tube of some stiffness but yet capable of being bent is needed. Drawn temper is for general purposes and is most commonly used where there is no specific requirement for high strength on the one hand

or for bending qualities on the other. Hard-drawn temper is used only where there is need for a tube as strong as is commercially feasible for the sizes indicated. For any combination of diameter and wall thickness not covered under hard-drawn temper, the values given for drawn temper shall be used.

Section 6.—Revise to read as follows:

6. The tempers of annealed tube shall be designated as light anneal and soft anneal (Table III).

Section 7.—Change the title to "Mechanical Properties of Drawn Temper Tube" and revise to read as follows:

7. Drawn temper tube shall conform to the mechanical properties prescribed in Table II. Tension tests are required for tubes under $\frac{5}{16}$ in. inside diameter or with a wall thickness under 0.020 in. The tension test for other sizes of tubes need not be made except when indicated by the purchaser at the time of placing the order. A convenient method of indicating that the tension test is required is to specify that "Test Procedure 'T' is required" (Section 3(*3*)). When agreement on the Rockwell hardness tests cannot be reached, the tensile strength requirements of Table II shall be the basis for acceptance or rejection.

Section 8.—Change the title to "Mechanical Properties of Annealed Temper Tube" and revise to read as follows:

8. Annealed temper tube shall conform to the grain size and Rockwell hardness limits prescribed in Table III.

Table II.—Revise to read as shown in the accompanying Table X.

Table III.—Revise to read as shown in the accompanying Table XI.

TABLE XI.—MECHANICAL PROPERTY REQUIREMENTS OF ANNEALED TEMPER TUBE.

(Revision of Table III, Specification B 135 - 55)

Alloy No.	(Annealed) Temper	Wall Thickness, in.	Rockwell Hardness[a]		Average Grain Size, mm	
			Scale	Max	Min	Max
1	Soft anneal.........	Up to 0.045 incl	30T	36	0.025	0.060
		Over 0.045	F	75	0.025	0.060
	Light Anneal.......	Up to 0.045 incl	30T	39	[b]	0.035
		Over 0.045	F	85	[b]	0.035
2, 3, and 4	Soft Anneal.........	Up to 0.030 incl	30T	40	0.025	0.060
		Over 0.030	F	80	0.025	0.060
2, 3, 4, 5 and 6	Light Anneal.......	Up to 0.030 incl	30T	60	[b]	0.035
		Over 0.030	F	90	[b]	0.035

[a] Rockwell hardness values shall apply only to tube having an inside diameter of $\frac{5}{16}$ in. or over and having a wall thickness of 0.015 in. or over. For all other tube no Rockwell values shall apply. Rockell hardness tests shall be made on the inside surface of the tube. When suitable equipment is not available for determining the specified Rockwell hardness, other Rockwell scales and values may be specified subject to agreement between purchaser and supplier.

[b] Although no minimum grain size is specified, the product must nevertheless have a fully recrystallized grain structure.

Standard Specification for Manganese Bronze Rod, Bar, and Shapes (B 138 - 54):

Section 7(a).—Add a Note to the second sentence to read "NOTE.—The tension test specimens shall conform to the dimensions prescribed in Sections 8, 10, 11, or 12 of Methods E 8 - 58 T."

Add a new sentence to read "Whenever tension test results are obtained from both full size and from machined test specimens and they differ, the results obtained from full size test specimens shall be used to determine conformance to the specification requirements."

Standard Specification for Phosphor Bronze Rod, Bar, and Shapes (B 139 – 52):

Section 6.—Add a Note to the second sentence to read "NOTE.—The tension test specimens shall conform to the dimensions prescribed in Sections 8, 10, 11, or 12 of Methods E 8 – 58 T."

Add a new sentence to read "Whenever tension test results are obtained from both full size and from machined test specimens and they differ, the results obtained from full size test specimens shall be used to determine conformance to the specification requirements."

Standard Specification for Leaded Red Brass (Hardware Bronze) Rod, Bar, and Shapes (B 140 – 54):

Table II.—Revise the last two lines of Table II so that all sizes of half-hard rectangles and squares have the following requirements:

Tensile strength, min, psi	40 000
Yield strength, min, psi	25 000
Elongation in 4× diameter or thickness of specimens, min, per cent	12

Section 6.—Following the second sentence add a note to read "NOTE.—The tension test specimens shall conform to the dimensions prescribed in Sections 8, 10, 11, or 12 of Methods E 8 – 58 T."

Add a new sentence to read "Whenever tension test results are obtained from both full size and from machined test specimens and they differ, the results obtained from full size test specimens shall be used to determine conformance to the specification requirements."

Standard Specification for Aluminum Bronze Rod, Bar, and Shapes (B 150 – 54):

Section 7(a).—Add a Note to the second sentence to read "NOTE.—The tension test specimens shall conform to the dimensions prescribed in Sections 8, 10, 11, or 12 of Methods E 8 – 58 T."

Add a new sentence to read "Whenever tension test results are obtained from both full size and from machined test specimens and they differ, the results obtained from full size test specimens shall be used to determine conformance to the specification requirements."

Standard Specification for Copper-Nickel-Zinc Alloy (Nickel Silver) Rod and Bar (B 151 – 55):

Section 7.—Add a note to the second sentence to read "NOTE.—The tension test specimens shall conform to the dimensions prescribed in Sections 8, 10, 11, or 12 of Methods E 8 – 58 T."

Add a new sentence reading "Whenever tension test results are obtained from both full size and from machined test specimens and they differ, the results obtained from full size test specimens shall be used to determine conformance to the specification requirements."

Standard Specification for Copper Sheet, Strip, Plate, and Rolled Bar (B 152 – 55):

Section 13.—Revise units of electrical resistivity to read "ohm—gram/meter^2."

Standard Method of Test for Expansion (Pin Test) of Copper and Copper-Alloy Tubing (B 153 – 55):

Section 2(a).—Replace the present Section 2(*a*) with the following:

> (*a*) *Pin.*—The pin shall have an included angle of 45 deg unless provided otherwise in the product specification. The pin shall be made of tool steel and shall be hardened and ground to the prescribed angle and shall have a smooth, polished surface. The size of the pin at the bar shall be suitable for the size of tubing being tested.

Standard Method of Mercurous Nitrate Test for Copper and Copper Alloys (B 154 - 51):

Section 1.—Add a new Note 3 to read as follows:

NOTE 3.—For any particular copper alloy, reference should be made to the material specification.

Section 3.—Add the following new sentence: "To obtain the most significant test on rod, wire, and tube, it is recommended that, in the absence of a prescribed specimen size in the product specification, the test specimen be at least 6 in. in length or twice the diameter, whichever is greater, when practicable."

Section 4.—Paragraph (*a*).—Revise the second sentence to read "If necessary totally immerse the specimen in an aqueous solution of sulfuric acid (15 per cent by volume) or nitric acid (40 per cent by volume) until all oxides are completely removed from its surface or pickle in such solutions as may be prescribed in the specification for the material being tested." In the fourth sentence add the words "at room temperature" following the words "totally immerse it."

Paragraph (*b*).—Add to the end of the third sentence "unless a time limitation is provided in the product specification."

Standard Specification for Phosphor Bronze Wire (B 159 - 57):

Table I.—Revise the tin limits under Alloy A from "3.5 to 5.8" to read "4.2 to 5.8."

Standard Specification for Copper-Alloy Condenser Tube Plates (B 171 - 55):

Section 7.—Change Footnote *b* under the tabulation of methods to read "[b] The tension test specimen shall conform to the dimensions shown in Figs. 7 or 8 of Methods E 8 - 58 T."

Standard Specification for Copper Bus Bar, Rod, and Shapes (B 187 - 55):

Section 8(*a*).—Add a Note to the second sentence to read "NOTE.—The tension test specimen shall conform to the dimensions prescribed in Sections 8, 10, 11, or 12 of Methods E 8 - 58 T."

Standard Specification for Seamless Copper Bus Pipe and Tube (B 188 - 56):

Section 2(*c*).—Revise to read as follows:

(*c*) *Average Diameter.*—The average outside or inside diameter of a tube is the average of the maximum and minimum outside diameters or the maximum and minimum inside diameters, whichever is applicable, as determined at any one cross-section of the tube.

Section 3.—Add a new item (*3*) to read as follows, renumbering subsequent items accordingly: "(*3*) Whether tension, elongation and bend tests are required (Section 5)."

Section 5.—Change the title to read "Mechanical and Electrical Properties." Revise Paragraph (*a*) to read as follows:

5. (*a*) The material shall conform to the mechanical and electrical resistivity or conductivity requirements prescribed in Table I. Tension, elongation, and bend tests need not be made except when indicated by the purchaser at the time of placing the order. A convenient method of indicating that these tests are required is to state that "Test Procedure 'T' is required" (Section 3 (*3*)). When agreement on the Rockwell hardness tests cannot be reached the tension test shall be made.

Table I.—Change the title to read "Mechanical and Electrical Property Requirements."

Section 10.—In the tabulation of tests in Paragraph (*a*), revise one line as follows:

Tension . . . E 8 (see also Section 10(*b*), (*c*), and (*d*)).

Renumber the present Paragraph (*b*) as (*e*) and add a new Paragraph (*b*) to read as follows:

(*b*) Tension test specimens shall be of the full section of the tube and shall conform to the requirements of Section 13(*a*) of ASTM Methods E 8, unless the limitations of the testing machine preclude the use of such a specimen. Test specimens conforming to type No. 1 of Fig. 13 (Section 13(*b*) of ASTM Methods E 8 may be used when a full section specimen cannot be tested).

Add new Paragraphs (*c*) and (*d*) to read as follows:

(*c*) Whenever tension test results are obtained from both full size and from machined test specimens and they differ, the results obtained from full size test specimens shall be used to determine conformance to the specification requirements.

(*d*) Tension test results on material covered by this specification are not seriously affected by variations in speed of testing. A considerable range of testing speed is permissible; however, the rate of stressing to the yield strength should not exceed 100,000 psi per min. Above the yield strength the movement per min of the testing machine head under load should not exceed 0.5 in. per in. of gage length (or distance between grips for full section specimens).

Section 12(d).—In the second line, change the word "physical" to read "mechanical and electrical."

Section 13.—Revise Paragraph (*j*) to read as follows:

(*j*) *Length for Water Tube.*—The standard length of copper water tube in straight lengths shall be 20 ft with a tolerance of 1 in., all plus.

Revise Paragraph (*k*) to read as follows:

(*k*) *Roundness.*—For drawn unannealed tube or pipe in straight lengths, the roundness tolerances shall be as follows:

t/D (Ratio of Nominal Wall Thickness to Nominal Outside Diameter)	Roundness Tolerance, as Per Cent of Nominal Outside Diameter (Expressed to the nearest 0.001 in.)
0.01 to 0.03 incl.	1.5
Over 0.03 to 0.05 incl.	1.0
Over 0.05 to 0.10 incl.	0.8 or 0.002 in., whichever is greater
Over 0.10	0.7 or 0.002 in., whichever is greater

Compliance with the roundness tolerance shall be determined by taking measurements on the outside diameter only, irrespective of the manner in which the tube dimensions are specified.

The deviation from roundness is measured as the difference between major and minor diameters as determined at any one cross-section of the tube.

No tolerances have been established for as-extruded tube, redrawn tube, annealed tube, any tube furnished in coils or drawn tube whose wall thickness is under 0.016 in.

Standard Specification for Silicon Bronze and Silicon Brass Sand Castings (B 198 – 52):

Section 6(d).—Revise the first sentence to read: "Unmachined specimens shall be cast in accordance with the requirements of Fig. 4, Specification B 208 – 54."

Recommended Practice for Tension Test Specimens for Copper-Base Alloys and Sand Castings (B 208 – 54):

Section 2.—Change paragraphs (*a*) and (*b*) to read as follows:

(*a*) The test bar coupons shall be made in the production sand in which the castings they represent are made wherever possible. Even if the castings are cast entirely in green sand, partial cores shall be permitted for the test bars, but in no case shall chills be permitted. Unless otherwise agreed upon by the manufacturer and purchaser, test bars may be poured in cores on a production line.

(*b*) *High-Shrinkage Alloys.*—The test specimen coupons for copper-base casting alloys exhibiting high shrinkage during freezing and cooling shall be cast to the form and dimensions shown in Fig. 1 or 3. If specimens having threaded ends are to be used in the tension tests, the length of the blocks in Fig. 1 may be reduced to 6¼ in.

Standard Classification of Coppers (B 224 – 52): Revise as appended hereto.[1]

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[1] The revised classification appears in the 1958 Book of ASTM Standards, Part 2.

Standard Specification for Threadless Copper Pipe (B 302 – 57):

Section 3.—Add a third item to read "(*3*) Whether tension tests are required (Section 6)."

Section 6.—Change the title to read "Mechanical Properties" and revise to read as follows:

6. (*a*) The material shall conform to the following mechanical property requirements:

Tensile strength, min, psi..........	36 000
Rockwell hardness, min, F scale....	55

When agreement on Rockwell hardness tests cannot be reached, the tensile strength requirements shall be the basis for acceptance or rejection.

(*b*) Tension tests need not be made except when indicated by the purchaser at the time of placing the order. A convenient method of indicating that this test is to be made is to state that "Test Procedure 'T' is required" (Section 3 (*3*)).

Section 7.—Revise the second sentence to read "The pipe need not be tested at a hydrostatic pressure of over 1000 psi unless so specified."

Section 9(c).—Change the title to read "Tests for Mechanical Properties." In the second line change "physical tests" to read "tests for mechanical properties."

Section 10.—Renumber the present Section 10 to 10(*a*). In the tabulation of tests, revise one line as follows:

Tension . . . E 8 (see also 10(*b*), (*c*) and (*d*) ff.)

Add new Paragraphs (*b*), (*c*), and (*d*) as follows:

(*b*) Tension test specimens shall be of the full section of the tube and shall conform to the requirements of Section 13(*a*) of ASTM Methods E 8, unless the limitations of the testing machine preclude the use of such a specimen. Test specimens conforming to type No. 1 of Fig. 13 (Section 13(*b*)) of ASTM Methods E 8 may be used when a full section specimen cannot be tested.

(*c*) Whenever tension test results are obtained from both full size and from machined test specimens and they differ, the results obtained from full size test specimens shall be used to determine conformance to the specification requirements.

(*d*) Tension test results on material covered by this specification are not seriously affected by variations in speed of testing. A considerable range of testing speed is permissible; however, the rate of stressing to the yield strength should not exceed 100,000 psi per minute. Above the yield strength the movement per minute of the testing machine head under load should not exceed 0.5 in. per in. of gage length (or distance between grips for full section specimens).

Section 13(e).—Revise the second sentence to read "The length tolerance shall be plus 1 in., minus 0 in."

Standard Specification for Copper Drainage Tube (DWV) (B 306 – 57):

Section 2.—Add a third item to read: "(*3*) Whether tension tests are required (Section 5)."

Section 5.—Change the title to read "Mechanical Properties" and revise to read as follows:

5. (*a*) The material shall conform to the following mechanical property requirements:

Tensile strength, min, psi..........	36 000
Rockwell hardness, min, F scale....	55

When agreement on Rockwell hardness tests cannot be reached, the tensile strength requirements shall be the basis for acceptance or rejection.

(*b*) Tension tests need not be made except when indicated by the purchaser at the time of placing the order. A convenient method of indicating that this test is to be made is to state that "Test Procedure 'T' is required" (Section 2(*3*)).

Section 6(c).—Change the title to read "Tests for Mechanical Properties" and revise the first sentence to read "The required tests for mechanical properties shall be made on each sample so selected."

Section 7.—Renumber the present Section 7 as Section 7(*a*). In the tabulation of tests, show the following change in one line:

Tension . . . E 8 (see also 7(*b*), (*c*) and (*d*) ff.)

Add new Paragraphs (*b*), (*c*) and (*d*) to read as follows:

(*b*) Tension test specimens shall be of the full section of the tube and shall conform to the requirements of Section 13(*a*), ASTM Methods E 8, unless the limitations of the testing machine preclude the use of such a specimen. Test specimens conforming to type No. 1 of Fig. 13 (Section 13(*b*)) of ASTM Methods E 8 may be used when a full section specimen cannot be tested.

(*c*) Whenever tension test results are obtained from both full size and from machined test specimens and they differ, the results obtained from full size test specimens shall be used to determine conformance to the specification requirements.

(*d*) Tension test results on material covered by this specification are not seriously affected by variations in speed of testing. A considerable range of testing speed is permissible; however, the rate of stressing to the yield strength should not exceed 100,000 psi per minute. Above the yield strength the movement per minute of the testing machine head under load should not exceed 0.5 in. per in. of gage length (or distance between grips for full section specimens).

Section 11(*e*).—Revise the second sentence to read "The length tolerance shall be plus 1 in., minus 0 in."

APPENDIX II

NUCLEAR RADIATION EFFECT ON COPPER AND COPPER ALLOYS*

By John Moteff[1]

The effects of nuclear radiation on materials, metals in particular, have been under extensive study since the construction of the first nuclear reactors. E. P. Wigner first called attention to the fact that energetic neutrons, born in a fission process, would have the ability to displace atoms from equilibrium positions in the crystal lattice of solids that might be far removed from the nuclear fuel, and as a consequence would have deleterious effects on many properties that would be of engineering interest in reactor construction.

Early experimental studies followed up by theoretical analysis by F. Seitz, G. J. Dienes, and H. Brooks confirmed Wigner's prophecy and led to suitable action by the reactor designer to allow for the damage expected to result from reactor operation. It soon became obvious that radiation damage was to be expected in all solid material and that the susceptibility of various materials to neutron and fission fragments effects would be a function of that particular solid. It was also apparent that the magnitude of the effect was a function of the neutron flux, the temperature, the environment during irradiation, and other variables such as stress.

Reactor Radiations

Figure 1 indicates the types of radiation that may be expected from a reactor. The three basic types of radiation resulting from fission are: (1) fission fragments, which are massive particles relative to the neutron and are highly charged. The damaging effects of these particles are generally confined to regions localized about the fuel elements. (2) Fast neutrons, which have a mass approximately equal to a hydrogen atom but have no electrical charge and hence are not subjected to forces due to the electrical fields present in interatomic distances. (3) Gamma rays, which behave similarly to X-rays. This radiation is not localized in any small area within the reactor, as are the fission fragments, but is essentially a diffuse distribution. An inspection of Fig. 1 will show that these gamma rays (which include *bremsstrahlung*) are also generated as a result of the nuclear processes and are characteristic of fission fragments and fast neutrons.

Figure 2 indicates the sensitivity of engineering properties of materials to radiation. This figure illustrates the fact that there are many types of damaging effects due to nuclear radiation on various materials. The radiation levels indicated are only approximate and subject to change as better experiments are performed. The indicated engineering property changes are in most cases at least 10 per cent. It should be pointed

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* This paper was presented at the Meeting of ASTM Committee B-5 on Copper and Copper Alloys, Cast and Wrought, held in Boston, Oct. 10, 1957.

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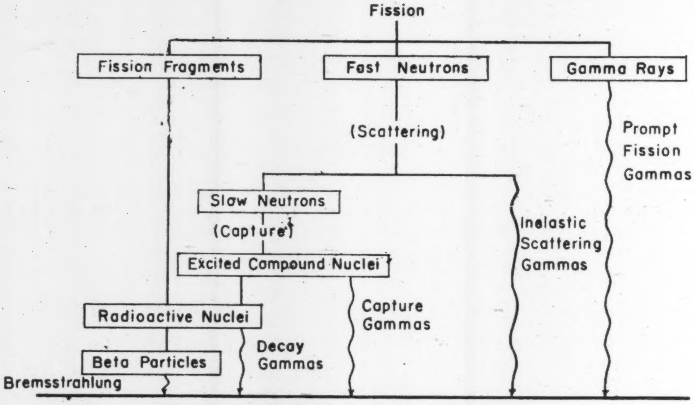
Fission
Fission Fragments
Fast Neutrons
Gamma Rays
(Scattering)
Prompt Fission Gammas
Slow Neutrons
(Capture)
Inelastic Scattering Gammas
Excited Compound Nuclei
Capture Gammas
Radioactive Nuclei
Decay Gammas
Beta Particles
Bremsstrahlung

FIG. 1.—Radiation from Nuclear Reactors.

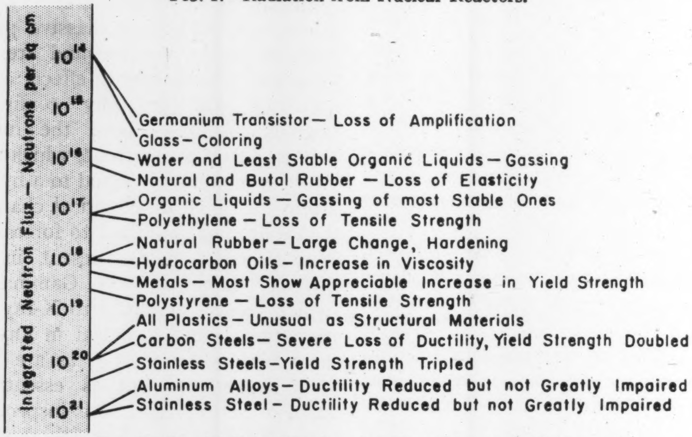

FIG. 2.—Sensitivity of Engineering Properties to Radiation.

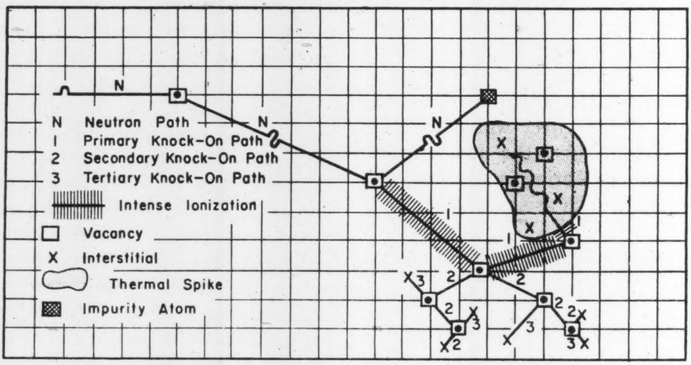
N Neutron Path
1 Primary Knock-On Path
2 Secondary Knock-On Path
3 Tertiary Knock-On Path
Intense Ionization
Vacancy
X Interstitial
Thermal Spike
Impurity Atom
N
1
2
3
X

FIG. 3.—Principal Mechanisms of Radiation Damage.

out that the magnitude of the irradiation dose indicated on the scale is in units of neutrons per sq cm. This general unit for describing the intensity of dosages is called integrated flux and is the product of the neutron flux (or gamma) and the time of exposure.

For instance, the Materials Testing Reactor has a fast neutron flux of about 3×10^{14} neutrons per sq cm per sec. This would mean that if ordinary glass were to be inserted in this reactor for approximately one third of a second it will undergo coloration. Natural rubber will start to lose elasticity when left in this reactor for roughly 30 sec. Metals begin to show some appreciable increase in yield strength when exposed for roughly 2 hr, whereas aluminum alloys and stainless steel begin to have their ductilities reduced, but not greatly impaired, when exposed for approximately 40 days.

MECHANISM OF RADIATION DAMAGE

Figure 3 illustrates the five principal mechanisms of radiation damage which are:

1. *Ionization.*—A process which results in the formation of ions. This effect is caused mostly by fission fragments, beta particles, and gamma rays in the fields of reactors.

2. *Vacancies.*—Sites in the crystal lattice of an ionic crystal from which the ions that should be present are missing. It is now reasonable to assume that the intermingling of atoms in the solid state depends on vacancies and that at any given temperature there is an equilibrium number of vacancies present; the larger the number of vacancies, the more rapidly diffusion takes place.

3. *Interstitials.*—Atoms occupying an interstitial position, that is, squeezed between the regular lattice sites in a crystal. The behavior of interstitials is more difficult to understand, but the pronounced increase in yield strength that accompanies even moderate neutron irradiation of metal leads one to believe that interstitials may be playing an important role in the hardening reactions.

4. *Impurity Atoms.*—Impurity atoms can be understood in conventional terms. Usually the number introduced due to neutron absorption is small, and the effect is small compared to other damage effects. In the matter of fission fragments, however, the impurity effect is of much larger magnitude.

5. *Thermal Spikes.*—The thermal spike presents the most controversial aspect of radiation damage. It results from that portion of the energy that is stored in the form of displaced atoms or trapped electrons and is dissipated as heat during the collision process. It has been generally supposed that this energy creates a momentary region of high temperature that in a good metal may involve thousands of atoms and result in an increase in temperature of 1000 Kelvin for probably less than 10^{-10} sec.

EFFECTS ON COPPER

Investigations into the effect of neutron irradiation on the mechanical properties of metals have up to now tended to fall into two vastly separate categories. The first of these are studies of the technically important material of nuclear reactor, for example, aluminum and magnesium alloys, steels, and recently developed metals such as zirconium, titanium, and columbium. Such materials are often complicated in behavior due to the complexity of their micro- and macrostructure and uncontrolled features such as unavoidable impurities. The second category which has been investigated to the largest extent is the behavior of single crystals under irradiation. Very little work has been carried out on the intermediate field of the effect of irradiation on polycrystalline metals of simple structure and well-known behavior. A few experiments have been performed by the British on the effects of neutron irradiation, and in particular the influence of testing temperature, on the mechanical properties of the face-centered cubic metal, copper.

Wire specimens were used. The specimens were irradiated for six months at about 100 C in a flux of 6×10^{12} slow

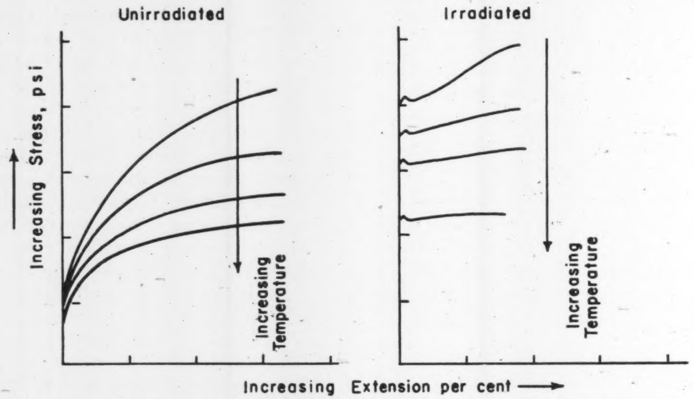

FIG. 4.—Stress-Strain Curves for Copper at Various Temperatures.

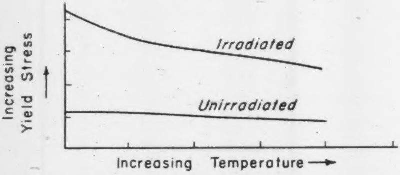

FIG. 5.—Effect of Irradiation on Yield Stress.

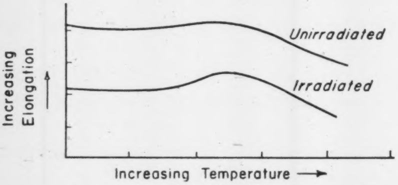

FIG. 6.—Effect of Irradiation on Elongation to Fracture.

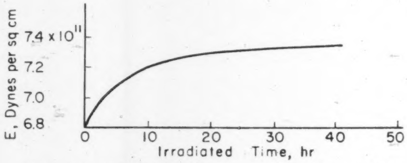

FIG. 7.—Effect of Gamma Irradiation on the Young's Modulus of Copper.

neutrons per sq cm per sec. The resulting total integrated flux of slow neutrons was of the order of 10^{19} neutrons per sq cm. An equal number of control specimens was prepared and stored at the same temperature and for the same time as the irradiation specimens. In this way, effects due to thermal history were eliminated. Some of the results of these tests are illustrated in Figs. 4, 5, 6, and 7.

The effects of radiation in mechanical properties of annealed copper were investigated over a wide temperature range in order to establish a general pattern of behavior.

Figure 4 shows the stress-strain curves for nonirradiated and irradiated copper. The behavior of the nonirradiated copper is in good agreement with published work on metal of similar purity.

The stress-strain curves for the irradiated specimens indicate that the yield stress is raised at all testing temperatures and that the stress-strain curves are no longer bunched together during the early stages of deformation. The rate of work hardening at low extensions is drastically reduced so that the curves at various temperatures are almost parallel. At large extensions, however, the curves still diverge with increasing strain. The elongation before fracture is appreciably less than before irradiation.

The effect of irradiation on yield stress is shown in greater detail in Fig. 5. The yield stress of the nonirradiated specimen shows only a slight temperature dependence. After irradiation, however, the yield stress is strongly dependent on the testing temperature, being much less at the higher temperatures. In the temperature range shown, the yield stress of irradiated copper decreased.

The effect of irradiation on the elongation before fracture is shown in Fig. 6. It is shown that the elongation before fracture is reduced by irradiation.

It should be pointed out that the curves used to illustrate these radiation effects are only approximate and should be considered only to indicate trends for these effects. If it is desired to have exact data, reference should be made to the specific articles.

Figure 7 indicates some of the other properties, such as the effect of gamma irradiation on the Young's modulus of copper. This experiment was performed on single crystals of copper subjected to irradiation with cobalt gamma rays at room temperature. This irradiation causes an increase in the Young's modulus and a decrease in the internal friction. Comparison with previous work shows that for each primary displaced lattice atom only $\frac{1}{80}$ as many pinning points results in the gamma ray bombardment as from bombardment with fast neutrons. These effects are quantitatively consistent with an interpretation on the basis of the pinning of dislocation lines by lattice defects which migrate to the dislocation line from the point at which they are originally produced through the Compton and photoelectron resulting from the incident gamma rays.

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† Compiled by P. K. Conn, Applied Radiation Effects Research Unit, Applied Materials Research Subsection, General Electric Co., Cincinnati, Ohio.

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(28) David K. Holmes and Donald O. Thompson, "Effects of Neutron Irradiation Upon the Young's Modulus and Internal Friction of Copper Single Crystals," *Journal of Applied Physics*, Vol. 27, July, 1953, p. 713.

(29) M. J. Makin, "The Effect of Neutron Irradiation on the Mechanical Properties of Metals—Part 3, Copper and Nickel," *Document No. AERE M/R 2080*, Atomic Energy Research Establishment, Harwell, Berkshire (England). (1956).

REPORT OF COMMITTEE B-6
ON
DIE-CAST METALS AND ALLOYS*

Committee B-6 on Die-Cast Metals and Alloys held two meetings during the year: in Atlantic City, N.J., on June 19, 1957, and in St. Louis, Mo., on February 12, 1958.

The committee consists of 92 members, of whom 41 are classified as producers, 21 as consumers and 30 as general interest members.

The officers and members-at-large on the Advisory Committee elected for the ensuing term of two years are as follows:

Chairman, W. Babington.

Vice-Chairman, A. E. Weiss.

Secretary, G. L. Werley.

Advisory Committee, Members-at-Large: F. C. Bennett, D. L. Colwell, R. F. Cramer, E. N. Jacobi, J. W. Meier, and A. J. Prickett.

REVISION OF TENTATIVE

The committee recommends revisions as follows of the Tentative Specification for Aluminum-Base Alloy Die Castings (**B 85 - 57 T**)[1] and continuation of the specification as tentative:

Table II.—Change ratings in the column headed "Resistance to Corrosion" to read as follows:

Alloy	Resistance to Corrosion
G 8A	1
S 5C	2
S 12A	3
S 12B	3
SC 84A	5
SC 84B	5
SG 100A	3
SG 100B	3
SC 114A	5

Appendix—Add a new Table III to the Appendix to read as shown in the accompanying Table I.

Replace the second paragraph of the Appendix with the following: "Table III shows the typical mechanical properties of test specimens cast in a test bar die and conforming to the chemical composition specified."

REVISION OF STANDARD, IMMEDIATE ADOPTION

The committee recommends for immediate adoption revision as follows of Standard Specifications for Magnesium-Base Alloy Die Castings (**B 94 - 57**),[1] and accordingly asks for the necessary nine-tenths affirmative vote at the Annual Meeting in order that the revision may be referred to letter ballot of the Society.

New Table.—Add a new Table II as shown in the accompanying Table II to replace the present unnumbered table in Note 1 of the Explanatory Notes.

EDITORIAL CHANGE

The committee recommends the following editorial change in the Standard Specification for Lead- and Tin-Base Alloy Die Castings (**B 102 - 52**):[2]

Table I.—In the first column remove the asterisk from alloy YC135A.

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[3]

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* Sixty-first Annual Meeting of the Society, June 22–27, 1958.

[1] 1957 Supplement to Book of ASTM Standards, Part 2.

[2] 1955 Book of ASTM Standards, Part 2.

[3] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

TABLE I.—TYPICAL MECHANICAL PROPERTIES OF TEST SPECIMENS.
(New Table III, Specification B 85 – 57 T)

Alloy	Tensile Strength, psi	Yield Strength (0.2 per cent Offset), psi	Elongation in 2 in., per cent	Shear Strength, psi	Fatigue Strength (R. R. Moore Specimen), 500,000,000 Cycles, psi
G 8A[a]	...	...	...	...	...
S 5C	33 000	14 000	9.0	19 000	17 000
S 12A	42 000	19 000	3.5	25 000	19 000
S 12B	43 000	21 000	2.5	25 000	19 000
SC 84A	47 000	23 000	3.5	27 000	20 000
SC 84B	46 000	23 000	2.5	28 000	20 000
SG 100A	46 000	24 000	3.5	26 000	18 000
SG 100B	44 000	25 000	2.5	28 000	20 000
SC 114A	48 000	24 000	2.5	29 000	20 000

[a] The mechanical property data for alloy G8A are in the process of being accumulated.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Aluminum-Base Die-Casting Alloys (E. V. Blackmun, chairman) prepared the revisions in Specification B 85 – 57 T covered earlier in this report.

The subcommittee is currently determining new mechanical properties for aluminum alloy G8A and investigating the advisability of recommending specification changes to recognize the commercial usage of the high zinc version of the SC84 alloy.

Subcommittee II on Zinc-Base Die-Casting Alloys (A. E. Weiss, chairman) is preparing to study the mechanical properties of zinc alloys cast in evacuated dies in comparison with the properties of regularly produced die-cast test bars.

Subcommittee V on Exposure and Corrosion Tests (D. H. Kleppinger, chairman) will recall zinc and magnesium alloy test bars from the exposure sites early next year after exposure for 20 yr. Volunteers will be accepted to conduct the mechanical testing program.

A progress report on exposure tests of aluminum alloys SG100A and G8A appears as an Appendix to this report.[4]

TABLE II.—TYPICAL PROPERTIES OF MAGNESIUM ALLOY DIE-CAST TEST SPECIMENS.
(New Table II, Specification B 94 – 57)

	Alloys AZ91A and AZ91B
Tensile strength, 1000 psi	32 to 36
Tensile yield strength, 1000 psi	22 to 24
Compression yield strength, 1000 psi	22 to 24
Elongation in 2 in., per cent	2 to 5
Impact, ft-lb[a]	2
Shear strength, 1000 psi[b]	20
Fatigue strength, 1000 psi[c]	14
Brinell hardness	63
Rockwell hardness, E scale	75

[a] Unnotched.
[b] Double shear tests converted to single shear value.
[c] 5×10^8 cycles.

Subcommittee VIII on Brass Die-Casting Alloys (G. F. Hodgson, chairman) reported that on the basis of recent data it seemed inadvisable to change the iron limit in alloy ZS331A from the present maximum of 0.15 per cent.

This report has been submitted to letter ballot of the committee, which consists of 92 members; 89 members returned their ballots, of whom 83 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

W. BABINGTON,
Chairman.

GEORGE L. WERLEY,
Secretary.

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[4] See p. 167.

APPENDIX

EXPOSURE TESTS OF ALUMINUM ALLOYS SG100A AND G8A

Progress Report of Subcommittee V on Exposure and Corrosion Tests

In 1943, Subcommittee I on Aluminum-Base Die-Casting Alloys planned a study of an 8 per cent magnesium alloy of aluminum and a simultaneous investigation of a 9 to 10 per cent silicon, 0.5 per cent magnesium alloy of aluminum. Aging tests of the latter were planned because of the "unknown aging effects of the magnesium content."[1] The 8 per cent magnesium alloy, now known as G8A, was added to ASTM Specifications B 85 in 1948,[2] and the 9.5 per cent silicon, 0.5 per cent magnesium alloy, now known as SG100A, first appeared in 1946.[3] Test bars were made by two producers, namely, Precision Castings Co. and Aluminum Co. of America, and exposed at two outdoor locations. One of these was on the seacoast (Sandy Hook, N. J.) and the other in an industrial atmosphere (New York, N. Y., on the roof of the Bell Laboratories Building). The reports of Committee B-6 for 1945, 1946, 1948, and 1953[4] report all progress up to the five years' atmospheric exposure. Many of the bars were lost before the completion of ten years, but a report on the 10-yr exposure was made to Subcommittee V on February 2, 1955. A summary of the exposure test results and also the initial accelerated aging test results is presented below. For the sake of continuity, comparison is also made, where applicable, with some of the work of the special Task Group on Properties which Subcommittee I has organized.

Alloy SG100A

It was decided to test two grades of alloy SG100A, one a commercial purity grade in accordance with ASTM Specifications B 85, and the other, a higher purity grade. As the 1948 report says, "Since the 9.5 per cent silicon, 0.5 per cent magnesium alloy was primarily intended as an alternate for the 12 per cent silicon alloy when cold chamber machines are to be used, test bars were produced only in cold chamber machines." Analyses of the two grades of SG100A and also of the SG100A which was tested by the Subcommittee I Task Group on Properties are given in Table I. These values are the averages of those from two laboratories, the variation between the two being less than experimental deviation.

Because of the concern held in 1943 that a magnesium silicide type of alloy might age harden considerably, shelf

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[1] Report of Committee B-6 on Die-Cast Metals and Alloys, *Proceedings*, Am. Soc. Testing Mats., Vol. 43, p. 173 (1943).

[2] Report of Committee B-6 on Die-Cast Metals and Alloys, *Proceedings*, Am. Soc. Testing Mats., Vol. 48, p. 191 (1948).

[3] 1946 Book of ASTM Standards, Part 1B, p. 670.

[4] Report of Committee B-6 on Die-Cast Metals and Alloys, *Proceedings*, Am. Soc. Testing Mats., Vol. 53, p. 207 (1953).

TABLE I.—AVERAGE CHEMICAL COMPOSITION OF SG100A TEST BARS FROM TWO PRODUCERS.[2]

(Cast in Cold Chamber Machine)

	Chemical Composition, per cent							
	Silicon	Iron	Copper	Magnesium	Manganese	Zinc	Nickel	Tin
SG100A[1] commercial purity	9.72	1.11	0.20	0.54	0.01	0.06	0.01	0.02
SG100A[1] high purity	9.61	0.30	0.04	0.54	<0.01	nil	<0.01	0.01
SG100A[5] 0.8 and 0.5 per cent iron)	9.38	1.00	0.49	0.52	0.25	0.42	0.05	—

TABLE II.—EFFECT OF INDOOR AGING AT ROOM TEMPERATURE: AVERAGE MECHANICAL PROPERTIES OF SG100A COMMERCIAL AND HIGH PURITY GRADES.[2]

	Commercial Purity				High Purity			
	Tensile Strength, psi	Yield Strength, psi	Elongation in 2 in., per cent	Charpy Impact, ft-lb	Tensile Strength, psi	Yield Strength, psi	Elongation in 2 in., per cent	Charpy Impact, ft-lb
As cast	43 700	25 800	3.7	2.7	43 800	24 300	5.5	4.2
Aged 3 months	42 900	26 100	4.0	2.8	43 100	24 700	4.9	4.4
Aged 6 months	46 200	27 400	3.3	2.7	43 500	24 500	5.2	4.4
Aged 12 months	46 200	27 400	3.0	2.6	43 400	24 800	4.3	4.3
Aged 24 months	45 900	27 300	2.8	2.7	44 400	25 200	4.3	4.0
Aged 29 months	43 800	28 400	2.7	na	45 700	26 000	6.0	na
Aged 33 months	44 700	28 300	2.9	na	45 400	25 800	4.9	na
Aged 42 months	45 700	27 900	3.3	na	44 500	25 800	4.7	na

TABLE III.—OUTDOOR ATMOSPHERIC EXPOSURE, AVERAGE OF NEW YORK AND SANDY HOOK LOCATIONS.

	Commercial Purity			High Purity		
	Tensile Strength, psi	Yield Strength, psi	Elongation in 2 in., per cent	Tensile Strength, psi	Yield Strength, psi	Elongation in 2 in., per cent
As cast[2]	43 700	25 800	3.7	43 800	24 300	5.5
Exposed 1 yr[2]	40 600	26 900	2.2	42 600	25 200	4.2
Exposed 2 yr[2]	41 600	26 800	2.2	42 300	25 000	3.8
Exposed 5 yr[4]	41 500	27 600	2.3	41 400	25 000	3.2
Exposed 10 yr[6]	39 600	27 600	1.6	41 400	25 400	3.5
Proposed specifications, 1957[5]	46 000	24 000	3.5			

TABLE IV.—20 PER CENT SALT SPRAY EXPOSURE AVERAGE.[2]

	Commercial Purity			High Purity		
	Tensile Strength, psi	Yield Strength, psi	Elongation in 2 in., per cent	Tensile Strength, psi	Yield Strength, psi	Elongation in 2 in., per cent
As cast	43 700	25 800	3.7	43 800	24 300	5.5
Exposed 12 weeks	39 500	26 100	2.4	38 300	26 200	2.0
Exposed 26 weeks	37 900	na	1.4	40 200	na	2.4
Exposed 52 weeks	35 300	25 500	1.6	39 000	25 400	2.7

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[5] Minutes of Subcommittee I on Aluminum-Base Die-Casting Alloys, June 19, 1957.

[6] Minutes of Subcommittee V on Exposure and Corrosion Tests, Feb. 2, 1955.

aging tests were conducted and the average results of this test are presented in Table II. The summary of the 10-yr atmospheric exposure tests is presented in Table III. Table III also includes the proposed properties for SG100A voted upon in 1957.

In addition to this, the tensile strengths and elongations following outdoor exposure are plotted in Fig. 1. Most of the figures are the values for two producers, tested by two laboratories, and the results averaged; in a few cases, only one laboratory participated. At the right of Fig. 1 are shown the tensile strength and elongation specification values proposed for specification in 1957.

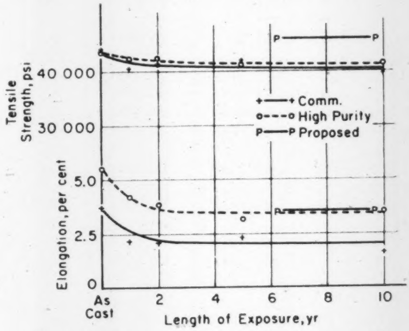

FIG. 1.—Effect of 10-yr Exposure of Alloy SG100A to Atmosphere.

A susceptibility of SG100A to age hardening is not shown in the 10-yr tensile strength curves of Fig. 1, but there is a considerable loss of elongation shown in both the commercial and high-purity grades over the 10-yr period. Part of this deterioration may have been due to corrosion effects. The 42-month indoor aging values of Table II were reasonably constant in both grades. The elongations of the high-purity grade were consistently higher than those of the commercial grade, and the yield strengths slightly lower. The tensile strength of 46,000 psi proposed seems high for either grade and the elongation of 3.5 per cent high for the commercial grade.

An accelerated corrosion test in 20 per cent salt spray was also conducted on alloy SG100A.[2] The summary of this test, averaging the values from two producers and two laboratories, is presented in Table IV, and the values are plotted in Fig. 2. Both tensile strengths and elongations fall somewhat faster than in atmospheric exposure because of the severe corrosion conditions, and again both grades of the alloy show similar effects of age hardening and corrosion. The higher elongations of the high-purity grade are evident and the loss in tensile strength of the commercial grade is slightly higher than that of the high-purity grade.

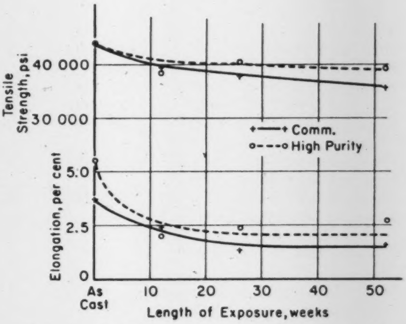

FIG. 2.—Effect of 1-yr Exposure of Alloy SG100A to 20 per cent Salt Spray.

ALLOY G8A

Similar tests were made on alloy G8A cast in both the gooseneck machine and the cold chamber machine by two producers. The compositions of these average test bars are given in Table V, and comparison is made with the average composition of bars of three iron contents, namely, 0.8 per cent, 1.2 per cent and 1.6 per cent, all cast in a cold cham-

TABLE V.—AVERAGE COMPOSITION OF G8A TEST BARS.

	Silicon, per cent	Iron, per cent	Copper, per cent	Magnesium, per cent	Manganese, per cent	Zinc, per cent
Cast in gooseneck machine[1]	0.21	1.06	0.04	7.86	0.03	0.05
Cast in cold chamber machine[1]	0.26	0.68	0.04	7.59	0.02	0.06
Average task group tests[7] (omitting 2 per cent iron)	0.33	1.22	0.22	8.10	0.22	0.11

TABLE VI.—EFFECT OF ATMOSPHERIC EXPOSURE ON THE MECHANICAL PROPERTIES OF G8A TEST BARS.

	Cast in Gooseneck Machine			Cast in Cold Chamber Machine		
	Tensile Strength, psi	Yield Strength, psi	Elongation in 2 in., per cent	Tensile Strength, psi	Yield Strength, psi	Elongation in 2 in., per cent
As cast[1]	37 000	24 600	4.5	41 200	25 100	7.4
(Producer B only)[1]	(40 000)	(25 500)	(5.2)	(44 600)	(27 200)	(9.3)
Exposed 1 yr New York[1]	38 400	25 800	4.2	42 900	26 200	7.5
Exposed 2 yr New York[1]	37 800	25 600	3.8	41 800	26 800	6.8
Exposed 5 yr New York[4]	38 400	26 800	3.8	41 500	26 200	5.2
Exposed 10 yr New York[6]	38 400	25 400	3.8	42 000	26 200	5.8
Exposed 1 yr Sandy Hook[2]	39 400	26 000	3.8	44 200	26 800	5.8
Exposed 2 yr Sandy Hook[2]	—	bars lost	—	—	bars lost	—
Exposed 5 yr Sandy Hook[4]	41 100	24 500	5.1	43 000	24 800	6.4
Exposed 10 yr Sandy Hook[6]	—	bars lost	—	—	bars lost	—
Average task group tests[5]	—	—	—	45 200	28 400	5.3

TABLE VII.—EFFECT OF EXPOSURE TO 20 PER CENT SALT SPRAY ON MECHANICAL PROPERTIES OF G8A TEST BARS.[2]

Exposure	Cast in Gooseneck Machine			Cast in Cold Chamber Machine		
	Tensile Strength, psi	Yield Strength, psi	Elongation in 2 in., per cent	Tensile Strength, psi	Yield Strength, psi	Elongation in 2 in., per cent
As cast	37 000	24 600	4.5	41 200	25 100	7.4
12 weeks	39 100	25 900	3.0	41 400	26 300	5.4
26 weeks	38 200	25 600	3.7	41 400	26 100	5.9
52 weeks	38 700	25 500	4.0	42 000	26 000	6.0

ber machine by three producers for the Task Group on Properties of Subcommittee I. These compositions are the averages of the analyses from two producers for each type machine and of the three compositions (except the 2 per cent iron) for the task group test. The 2 per cent iron series of the latter was omitted for more accurate comparison, and the three compositions of the task group test were almost identical except for the iron. While all compositions shown in Table V are within the limits of ASTM Specifications B 85, it will be noted that the task group test bars were noticeably higher in silicon, iron, copper, and magnesium.

In view of the tendency of magnesium

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[7] Letter reporting ingot analyses to task group, March 15, 1957.

to oxidize, it was considered advisable to hold the magnesium alloy at casting temperature for 8 hr and to test sample test bars each hour. This was done by both producers in both types of machines and tested by two laboratories; the resulting values are given in the 1948 report of Committee B-6.[2] No significant change could be noted over the 8-hr period, and the bars made by the cold chamber process were considerably stronger in both tensile strength and elongation than those made in the gooseneck machine. In this alloy, the bars made by producer B had significantly higher values in tensile strength, yield strength, elongation, and Charpy impact strength than did those made by producer A. The results of the room temperature aging tests gave a similar trend, with perhaps an increase in the tensile and yield properties during the first 26 months, with constant properties thereafter. Further details are given in the 1948 report.

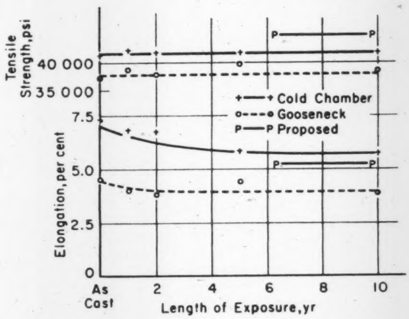

FIG. 3.—Effect of 10-yr Exposure of Alloy G8A to Atmosphere.

Both types of bars were exposed out of doors at the same two locations, namely, New York City and Sandy Hook, N. J., for 10 yr. The average values of this test from both producers are given in Table VI, but here the New York values and the Sandy Hook values are given separately because the bars for the 2-yr and 10-yr tests at Sandy Hook were lost. These tensile strengths and elongations are plotted in Fig. 3, averaging the values for the two locations at the start, 1-yr and 5-yr points, and using only the New York values at the 2-yr and 10-yr points because of the lost bars at Sandy Hook. In spite of this inconsistency, the curves of Fig. 3 are smooth and agree with the general shape of the salt spray corrosion curves of Fig. 4. There was no significant change in tensile strength; cold chamber values were higher than gooseneck; and there was a slight falloff in elongation, particularly in the higher cold chamber values. The tensile strength and elongation values of the work of Task Group on Properties are also shown in Table VI and at the right of Fig. 3 for comparison. Because the initial values of producer B were noticeably higher than those of producer A these initial values are shown in Table VI in parentheses, but the trends were the same for both producers. In these outdoor exposure tests alloy G8A was practically immune to deterioration.

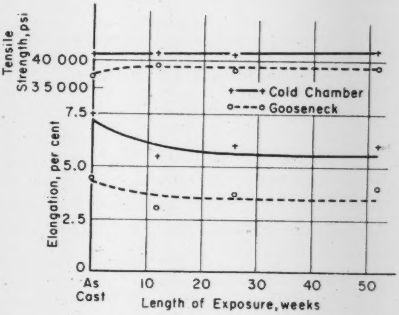

FIG. 4.—Effect of 1-yr Exposure of Alloy G8A to 20 per cent Salt Spray.

An accelerated corrosion test was also conducted on alloy G8A on bars from both gooseneck and cold chamber machines.[2] The summary of this test is presented in Table VII, again averaging the values from two producers and two laboratories. The plot is shown in Fig. 4. This severe test again proved the superior corrosion resistance of alloy G8A as the tensile strengths of bars from both machines remained constant, although the cold chamber bars were slightly stronger. The effects of corrosion on elongation agreed with the effects of the 10-yr exposure; both types of bars lost very little elongation, and the cold chamber bars were at a higher level.

COMPARISON OF ALLOYS

The indoor shelf aging properties of both alloys showed no appreciable changes in properties in either alloy in either condition.[2] The tensile strengths, yield strengths, and elongations for both types of alloy SG100A and for both machines of alloy G8A for 24 months for the former and 26 months for the latter are tabulated in Table VIII, together with the proposed specification values for alloy SG100A and the task group results of alloy G8A for only the 1.2 per cent iron content. The latter are the averages from three producers and the former are averages of two producers who participated in both tests.

In all tables, the tensile strengths and yield strengths are indicated in psi and the elongations in per cent in 2 in., all obtained on the standard ASTM $\frac{1}{4}$ in. round bar. Where impact values are shown, they were obtained upon the Charpy machine, on the standard ASTM $\frac{1}{4}$ in. square bar and are indicated in ft-lb. The curves of Figs. 1 through 4 are intended to present qualitative comparisons; because all four are on the same scale, comparisons between figures can be made qualitatively.

TABLE VIII.—COMPARISON OF PROPERTIES.

	Tensile Strength, psi	Yield Strength, psi	Elongation in 2 in., per cent	Report
SG100A—Commercial (24 months)	45 900	27 300	2.8	1948[2]
SG100A—High purity (24 months)	44 400	25 200	4.3	1948[2]
SG100A—1.2 per cent iron, Subcommittee I	47 600	25 700	3.3	1957[5]
SG100A—For specification	46 000	24 000	3.5	1957[5]
G8A—Cold chamber (26 months)	44 400	26 600	7.0	1948[2]
G8A—Gooseneck (26 months)	38 800	26 000	3.6	1948[2]
G8A—1.2 per cent iron, Subcommittee I	45 700	29 000	5.0	1957[5]

Respectfully submitted by the Task Group,

D. L. COLWELL, *Chairman.*
E. V. BLACKMUN and
D. H. KLEPPINGER.

REPORT OF COMMITTEE B-7

ON

LIGHT METALS AND ALLOYS, CAST AND WROUGHT*

Committee B-7 on Light Metals and Alloys held two meetings during the year: at Atlantic City, N. J., in June 1957, and at St. Louis, Mo., in February 1958.

The committee consists of 88 members, of whom 85 are voting members; 37 are classified as producers, 32 as consumers, and 19 as general interest members.

Mr. Keller resigned as chairman of Subcommittee VI and was replaced by R. F. Hafer. H. R. Youngkrantz passed away and W. E. Day, Jr. and E. H. Holzworth resigned from the committee because of retirement.

Two new subcommittees were organized: Subcommittee IX, Editorial, R. A. Harris, chairman; and Special Subcommittee on Light Metals for Nuclear Applications, W. G. Groth, chairman.

The Advisory Committee functioned as the advisory group to the American Standards Assn. which represents the United States as a participating member of Technical Committee 79 on Light Metals and Their Alloys of the International Organization for Standardization (ISO).

The officers elected for the ensuing term of two years are as follows:

Chairman, I. V. Williams.
Vice-Chairman, D. L. Colwell.
Vice-Chairman, C. B. Gleason.
Secretary, R. B. Smith.

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1957 Annual Meeting, the committee presented to the Society through the Administrative Committee on Standards a revision of Tentative Specification for Aluminum-Alloy Round Welded Tubes (B 313 – 56 T). The revision was accepted by the Administrative Committee on Standards on November 1, 1957, and it appears in the 1957 Supplement to Book of ASTM Standards, Part 2.

RECOMMENDATIONS AFFECTING STANDARDS

The committee recommends one new specification for publication as tentative, revisions in 17 tentative specifications, adoption of 6 tentatives as standard with revision, adoption of one tentative as standard without revision, revision of 2 standards for immediate adoption, and reapproval of 3 standards. The standards and tentatives affected, together with the revisions recommended, are covered in detail in Appendix I of this report.[1]

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[2]

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* Sixty-first Annual Meeting of the Society, June 22–27, 1958.

[1] See p. 177.

[2] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Aluminum and Aluminum Alloy Ingots (R. A. Quadt, chairman) recommended the new Tentative Specification for Aluminum-Alloy Hardeners for Making Zinc-Base Die-Casting Alloys; and revisions in the Standard Specification for Aluminum-Base Alloys in Ingot Form for Sand Castings, Die Castings, and Permanent Mold Castings (B 179 – 57) including the deletion of alloy SC54A-B and the addition of alloy GM70B, addition of new sections on basis of purchase and marking for identification, and an appendix to the specification showing the color codes referred to in the new section. All changes are set forth in Appendix I of this report.

Subcommittee II on Aluminum Alloy Castings (D. L. Colwell, chairman) prepared the revision of the Tentative Specification for Aluminum-Base Alloy Sand Castings (B 26 – 57 T) and Tentative Specification for Aluminum-Base Permanent Mold Castings (B 108 – 57 T) as set forth in the Appendix to this report. These changes add an alloy, GM70B, to both specifications; replace alloys SC64B and C in Tables I, II, and IV with SC64D in Specification B 26; and replace alloys SC64A, B, and C in Tables I, II, and IV with Alloy SC64D in Specification B 108.

Subcommittee III on Wrought Aluminum and Wrought Aluminum Alloys (H. D. Monsch, chairman) revised Tentative Specifications B 209, B 210, B 211, B 221, B 234, B 235, B 236, B 241, B 247, B 307, B 308, B 313, B 316, B 317, and B 318, as set forth in the Appendix to this report, to add a column giving the AA alloy designation corresponding to the ASTM alloy designation in tabular data and a footnote reference to ASA Standard H35.1—1957 which covers the AA alloy designation system, with the exception of B 236.

In addition, changes are recommended in Specification B 209 to revise chemical requirements for alloys GM40A, GM41A, and GM51A; also to add chemical and tensile requirements for alloys GM31A, GR20B, and GR40B.

Changes in Specifications B 210, B 234, B 235, and B 241 were made to clarify the heading of the column for elongation in Table II by adding a footnote; also in B 234 and B 307 to clarify the flattening test in Section 13.

Changes in Specifications B 210, B 211, B 221, and B 235 to add chemical and tensile requirements for alloys GR20B and GR40B in each case where requirements for alloy GR20A and GR40A appear in the 1957 revision of these specifications. Chemical and tensile requirements have also been added for alloy clad GM50A wire to Specification B 211, and for alloy GM31A to Specifications B 221, B 235, B 241, and B 308.

Changes in Specifications B 235 and B 241 are recommended to add chemical requirements and tensile requirements for alloy GM51A.

Changes in Specification B 247 are recommended to revise the temper designations for alloys 990A and M1A in Table II.

Changes are recommended in Specifications B 221 and B 308 to add chemical requirements and tensile requirements for alloys GM40A and GM41A; also in B 308 to add tensile requirements for GS11C-T62.

Editorial changes are recommended in Section 6 of Specification B 236 and in Tables IV and VII of Specification B 317; also Section 9(*c*) of B 236 to permit use of the eddy current method for determination of conductivity.

Subcommittee IV on Magnesium and Magnesium Alloys, Cast and Wrought (A. A. Moore, chairman) revised Specifications B 80 – 57 T, B 91 – 57 T, B 93 – 57 T, B 107 – 57 T, B 199 – 57 T, and

B 217 - 57 T as set forth in the Appendix of this report. All of the above specifications and Specification B 90 - 57 T are recommended for adoption as standards.

Specifications B 80 - 57 T and B 199 - 57 T were revised to include minimum yield strength requirements on all alloys and conditions. In Specifications B 91 - 57 T, B 107 - 57 T, and B 217 - 57 T the silicon was lowered from 0.30 per cent max to 0.10 per cent max on alloys where it is shown. The calcium in alloy AZ31B was specified as 0.04 per cent max in Specifications B 91 - 57 T, B 107 - 57 T, and B 217 - 57 T.

Subcommittee V on Testing Light Metals (J. C. Millson, chairman) proposed revisions to Specifications B 234 - 57 T and B 307 - 57 T to correct controlling dimension in tube flattening test to the maximum, not the minimum, and to Specifications B 210 - 57 T, B 234 - 57 T, B 235 - 57 T, and B 241 - 57 T to add a Note to Table II to clarify the gage length for determining per cent elongation.

The subcommittee is holding open the question of the establishment of lot sizes for statistically based sampling plans for tensile requirements of pertinent specifications, pending advice from Committee E - 11 on Quality Control of Material.

A task group is working to establish suitable testing speeds for short-time elevated-temperature tension tests, and is also cooperating with the Test Methods Panel of the Joint ASME-ASTM Committee on the Effect of Temperature on the Properties of Metals.

A task group is also dealing with the problem of the satisfactory minimum size of subsize tension test bars, including rounds and flats, both cast and wrought.

Consideration is being given to expressing stress values in terms of thousands of pounds per square inch.

Subcommittee VI on Anodic Oxidation of Aluminum and Magnesium Alloys (R. F. Hafer, chairman) recommends that Standard Methods of Test for Dielectric Strength of Anodically Coated Aluminum (B 110 - 45), Sealing of Anodically Coated Aluminum (B 136 - 45), and Weight of Coating on Anodically Coated Aluminum (B 137 - 45) be reapproved since they do not require revision.

A proposed Tentative Specification for Anodic Coatings on Aluminum and Aluminum Alloys has been completed with the exception of an appendix.

Subcommittee VII on Codification of Light Metals and Alloys, Cast and Wrought (C. B. Gleason, chairman) revised Recommended Practice for Codification of Light Metals and Alloys, Cast and Wrought (B 275 - 55) to provide a symbol for lithium, preliminary assignment of designations, and designations for granular metal products as set forth in the Appendix to this report.

Subcommittee VIII on Atmospheric Exposure Tests (L. H. Adam, chairman) has test specimens of aluminum and magnesium alloys exposed for over five years at five ASTM atmospheric exposure test sites. Data obtained from cast specimens exposed for three years are appended to this report.[3]

Subcommittee IX on Editorial (R. A. Harris, chairman) was organized with membership appointed from each of the other subcommittees of Committee B-7. The subcommittee approved the adoption of Proposed Recommendations on Form of ASTM Specifications, April 1957, as a guide, insofar as practical, for the editorial practices applying to Committee B-7 specifications. A task group has been appointed to prepare additional supplementary recommendations for editorial practices applying particularly to Committee B-7 specifications.

Special Subcommittee on Light Metals

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[3] See p. 194.

for Nuclear Applications (W. G. Groth, chairman) has been organized to guide the other subcommittees in writing specifications for light metals for nuclear applications. A paper on "Applications of Aluminum and Magnesium in the Nuclear Industries" by the subcommittee chairman is appended to this report.[4]

This report has been submitted to letter ballot of the committee, which consists of 88 members; 59⅔ members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

I. V. WILLIAMS,
Chairman.

R. B. SMITH,
Secretary.

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[4] See p. 210.

APPENDIX I

PROPOSED RECOMMENDATIONS AFFECTING STANDARDS FOR LIGHT METALS AND ALLOYS, CAST AND WROUGHT

In this Appendix are given the recommendations affecting certain standards and tentatives covering light metals and alloys which are referred to earlier in this report. The standards and tentatives appear in their present form in the 1955 Book of ASTM Standards, Part 2, or in the 1956 and 1957 Supplements to Book of ASTM Standards, Part 2.

All of the specifications covering wrought aluminum and aluminum alloys are having their tables revised to include the AA alloy designations along with the ASTM designations. Table I lists the corresponding alloys. In each of the revisions for the wrought product specifications the appropriate AA designation will be listed along with the presently listed ASTM designation.

New Tentative

The committee recommends for publication as tentative the Specification for Aluminum Alloy Hardeners Used in Making Zinc-Base Die Casting Alloys as appended hereto.[1]

Revisions of Tentatives

The committee recommends revisions as indicated of the following tentatives and their continuation as tentative:

Tentative Specification for Aluminum-Base Alloy Sand Castings (B 26 – 57 T):

Table I.—Add to the table alloy GM70B, with the chemical composition shown below. Delete alloys SC64B and SC64C and replace with SC64D, with the chemical requirements as shown:

	Aluminum	Copper	Iron	Silicon	Manganese	Magnesium	Zinc	Chromium	Titanium	Nickel	Other Elements	
											Each	Total
GM70B...	remainder	0.10	0.25[f]	0.20[f]	0.10 to 0.25	6.2 to 7.5	...	...	0.10 to 0.25	...	0.05	0.15
SC64D....	remainder	3.5 to 4.5	1.0	5.5 to 7.0	0.50	0.10	1.0	...	0.25	0.35	...	0.50

[f] Iron plus silicon not to exceed 0.40.

Table II.—Add to the table alloy GM70B, with the tensile requirements shown below. Delete alloys SC64B and SC64C and replace with SC64D, with the tensile requirements as shown:

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[1] The new tentative appears in the 1958 Book of ASTM Standards, Part 2.

	Condition	Yield Strength (0.2 per cent offset), min, psi	Tensile Strength, min, psi	Elongation in 2 in., min, per cent
GM70B	...	18 000	35 000	9.0
SC64D	F	13 000	23 000	1.5
	T6	20 000	32 000	2.5

TABLE I.—AA AND ASTM ALLOY DESIGNATIONS.

ASTM Designation	AA Designation[a]
990A	1100
996	1060
CG30A	2117
CG42A	2024
Clad CG42A	Alclad 2024
CB60A	2011
CM41A	2017
CN42C	2018
CN42D	2218
CS41A	2014
Clad CS41A	Alclad 2014
CS41C	2025
G1A	5050
G1B	5005
GM31A	5454
GM40A	5086
GM41A	5083
GM50A	5056
Clad GM50A	Alclad 5056
GM51A	5456
GR20A	5052
GR20B	5652
GR40A	5154
GR40B	5254
GS10A	6063
GS11A	6061
Clad GS11A	Alclad 6061
GS11B	6053
GS11C	6062
M1A	3003
Clad M1A	Alclad 3003
MG11A	3004
Clad MG11A	Alclad 3004
SG11A	6151
SG11B	6066
SG121A	4032
ZG62A	7075
Clad ZG62A	Alclad 7075

[a] These alloy designations are established in accordance with ASA Standard H35.1—1957.

Table IV.—Add to the table alloy GM70B, with the properties and characteristics shown in the accompanying Table II. Delete alloys SC64B and SC64C and replace with SC64D, with the properties and characteristics shown in Table II.

TABLE II.— ADDITIONS TO TABLE IV, SPECIFICATION B 26 - 57 T.

	GM70B	SC64D
Pattern shrinkage	5/32	5/32
Melting range	1020 to 1165	950 to 1125
Resistance to hot cracking	3	2
Pressure tightness	5	2
Fluidity	5	2
Solidification shrinkage tendency	5	2
Normally heat-treated	no	yes
Resistance to corrosion	1	3
Machining	1	3
Polishing	1	4
Electroplating	5	2
Anodizing (appearance)	1	4
Chemical oxide coating (protection)	1	3
Strength at elevated temperatures	3	3
Suitability for welding	4	2
Suitability for brazing	no	no

Tentative Specification for Aluminum-Base Alloy Permanent Mold Castings (B 108 - 57 T):

Table I.—Add to the table alloy GM70B, with the chemical composition as indicated above for Specification B 26 - 57 T. Delete alloys SC64A, SC64B, and SC64C and replace with SC64D, with the chemical requirements as indicated above for Specification B 26 - 57 T.

Table II.—Add to the table alloy GM70B, with the tensile requirements shown below. Delete alloys SC64A, SC64B, and SC64C, and replace with SC64D, with the tensile requirements as shown:

	Condition	Yield Strength (0.2 per cent offset), min, psi	Tensile Strength, min, psi	Elongation in 2 in., min, per cent
GM70B...	...	18 000	35 000	8.0
SC64D...	F.....	14 000	27 000	2.5
	T61...	24 000	40 000	2.0

TABLE III.—ADDITIONS TO TABLE IV, SPECIFICATION B 108 - 57 T.

	GM70B	SC64D
Melting range..............	1020 to 1165	950 to 1125
Resistance to hot cracking.....	4	2
Pressure tightness...........	5	2
Fluidity.....................	5	2
Solidification shrinkage tendency.....................	5	3
Normally heat treated........	no	yes
Resistance to corrosion........	1	3
Machining...................	1	3
Polishing....................	1	3
Electroplating...............	5	2
Anodizing (appearance).......	1	4
Chemical oxide coating (protection)..................	1	3
Strength at elevated temperature......................	3	3
Suitability for welding........	4	2
Suitability for brazing........	no	no

Table IV.—Add to the table alloy GM70B, with the properties and characteristics shown in the accompanying Table III. Delete alloys SC64A, SC64B, and SC64C and replace with SC64D, with the properties and characteristics shown in Table III.

Tentative Specification for Aluminum Alloy Sheet and Plate (B 209 - 57 T):

Table I.—Add a column presenting the AA alloy designation corresponding to the ASTM alloy designation, as shown in Table I; also add a new Footnote *b* referenced to the column heading "AA Designation," renumbering subsequent footnotes accordingly, to read: "[b] These AA alloy designations of the Aluminum Association were established in accordance with the American Standard Alloy Designation System for Wrought Aluminum (ASA No. H35.1—1957)."

Revise the chemical requirements for alloys GM40A, GM41A, and GM51A, and add chemical requirements for alloys GM31A, GR20B, and GR40B; also add the AA alloy designation correspond-

TABLE IV.—ADDITIONS TO TABLE I, SPECIFICATION B 209 - 57 T.

Alloy		Aluminum, per cent	Copper, per cent	Iron, per cent	Silicon, per cent	Manganese, per cent	Magnesium, per cent	Zinc, per cent	Chromium, per cent	Titanium, per cent	Beryllium, per cent	Other Elements, per cent	
ASTM Designation[a]	AA Designation[b]											Each	Total
GM31A....	5454...	remainder	0.10	[g]	[g]	0.50 to 1.0	2.4 to 3.0	0.25	0.05 to 0.20	0.20	...	0.05	0.15
GM40A....	5086...	remainder	0.10	0.50	0.40	0.20 to 0.7	3.5 to 4.5	0.25	0.05 to 0.25	0.15	...	0.05	0.15
GM41A....	5083...	remainder	0.10	0.40	0.40	0.30 to 1.0	4.0 to 4.9	0.25	0.05 to 0.25	0.15	...	0.05	0.15
GM51A....	5456...	remainder	0.20	[g]	[g]	0.50 to 1.0	4.7 to 5.5	0.25	0.05 to 0.20	0.20	0.0005	0.05	0.15
GR20B....	5652...	remainder	0.04	[f]	[f]	0.01	2.2 to 2.8	0.20	0.15 to 0.35	...	...	0.05	0.15
GR40B....	5254...	remainder	0.05	[f]	[f]	0.01	3.1 to 3.9	0.20	0.15 to 0.35	0.05	...	0.05	0.15

TABLE V.—ADDITIONS TO TABLE II, SPECIFICATION B 209 - 57 T.

Temper	Thickness, in.	Tensile Strength, psi		Yield Strength, psi		Elongation in 2 in., min, per cent	Bend Diameter Factor, *N*
		Min	Max	Min	Max		
ASTM ALLOY GM31A OR AA ALLOY 5454							
O	0.020 to 0.031	31 000	41 000	12 000[h]	...	12	...
	0.032 to 0.050	31 000	41 000	12 000[h]	...	14	...
	0.051 to 0.113	31 000	41 000	12 000[h]	...	16	...
	0.114 to 3.000	31 000	41 000	12 000[h]	...	18	...
H32	0.020 to 0.050	36 000	44 000	26 000[h]	...	5	...
	0.051 to 0.249	36 000	44 000	26 000[h]	...	8	...
	0.250 to 2.000	36 000	44 000	26 000[h]	...	12	...
H34	0.020 to 0.050	39 000	47 000	29 000[h]	...	4	...
	0.051 to 0.161	39 000	47 000	29 000[h]	...	6	...
	0.162 to 0.249	39 000	47 000	29 000[h]	...	7	...
	0.250 to 1.000	39 000	47 000	29 000[h]	...	10	...
H112	0.250 to 0.499	32 000	...	18 000[h]	...	8	...
	0.500 to 2.000	31 000	...	12 000[h]	...	11	...
	2.001 to 3.000	31 000	...	12 000[h]	...	15	...
ASTM ALLOY GR20B OR AA ALLOY 5652							
O	0.006 to 0.007	25 000	31 000	...	...	...	...
	0.008 to 0.019	25 000	31 000	...	...	15	...
	0.020 to 0.031	25 000	31 000	...	...	18	...
	0.032 to 0.050	25 000	31 000	...	...	20	...
	0.051 to 0.249	25 000	31 000	9 500[h]	...	20	...
	0.250 to 3.000	25 000	31 000	9 500[h]	...	18	...
H32	0.017 to 0.019	31 000	38 000	...	...	4	...
	0.020 to 0.050	31 000	38 000	...	...	5	...
	0.051 to 0.113	31 000	38 000	23 000[h]	...	7	...
	0.114 to 0.249	31 000	38 000	23 000[h]	...	9	...
	0.250 to 0.500	31 000	38 000	23 000[h]	...	11	...
	0.501 to 2.000	31 000	38 000	23 000[h]	...	12	...
H34	0.009 to 0.019	34 000	41 000	...	...	3	...
	0.020 to 0.050	34 000	41 000	...	...	4	...
	0.051 to 0.113	34 000	41 000	26 000[h]	...	6	...
	0.114 to 0.249	34 000	41 000	26 000[h]	...	7	...
	0.250 to 1.000	34 000	41 000	26 000[h]	...	10	...
H36	0.006 to 0.007	37 000	44 000	...	...	...	...
	0.008 to 0.031	37 000	44 000	...	...	3	...
	0.032 to 0.162	37 000	44 000	...	...	4	...
H38	0.006 to 0.007	39 000	...	...	...	...	...
	0.008 to 0.031	39 000	...	...	...	3	...
	0.032 to 0.128	39 000	...	...	...	4	...
H112	0.250 to 0.500	28 000	...	16 000[h]	...	7	...
	0.501 to 2.000	25 000	...	9 500[h]	...	12	...
	2.001 to 3.000	25 000	...	9 500[h]	...	16	...
ASTM ALLOY GR40B OR AA ALLOY 5254							
O	0.020 to 0.031	30 000	41 000	11 000[h]	...	12	...
	0.032 to 0.050	30 000	41 000	11 000[h]	...	14	...
	0.051 to 0.113	30 000	41 000	11 000[h]	...	16	...
	0.114 to 3.000	30 000	41 000	11 000[h]	...	18	...

TABLE V.—*Continued*

Temper	Thickness, in.	Tensile Strength, psi		Yield Strength, psi		Elongation in 2 in., min, per cent	Bend Diameter Factor, N
		Min	Max	Min	Max		
ASTM ALLOY GR40B OR AA ALLOY 5254—*Continued*							
H32	0.020 to 0.050	36 000	43 000	26 000[h]	...	5	...
	0.051 to 0.249	36 000	43 000	26 000[h]	...	8	...
	0.250 to 2.000	36 000	43 000	26 000[h]	...	12	...
H34	0.020 to 0.050	39 000	46 000	29 000[h]	...	4	...
	0.051 to 0.161	39 000	46 000	29 000[h]	...	6	...
	0.162 to 0.249	39 000	46 000	29 000[h]	...	7	...
	0.250 to 1.000	39 000	46 000	29 000[h]	...	10	...
H36	0.020 to 0.050	42 000	49 000	32 000[h]	...	3	...
	0.051 to 0.113	42 000	49 000	32 000[h]	...	4	...
	0.114 to 0.162	42 000	49 000	32 000[h]	...	5	...
H38	0.020 to 0.050	45 000	...	35 000[h]	...	3	...
	0.051 to 0.113	45 000	...	35 000[h]	...	4	...
	0.114 to 0.128	45 000	...	35 000[h]	...	5	...
H112	0.250 to 0.500	32 000	...	18 000[h]	...	8	...
	0.501 to 2.000	30 000	...	11 000[h]	...	11	...
	2.001 to 3.000	30 000	...	11 000[h]	...	15	...

TABLE VI.—ADDITION TO TABLE I, SPECIFICATION B 210 - 57 T.

Alloy		Aluminum, per cent	Copper, per cent	Iron, per cent	Silicon, per cent	Manganese, per cent	Magnesium, per cent	Zinc, per cent	Chromium, per cent	Titanium, per cent	Other Elements, per cent	
ASTM Designation[a]	AA Designation[b]										Each	Total
GR20B	5652	remainder	0.04	[d]	[d]	0.01	2.2 to 2.8	0.20	0.15 to 0.35	...	0.05	0.15
GR40B	5254	remainder	0.05	[d]	[d]	0.01	3.1 to 3.9	0.20	0.15 to 0.35	0.05	0.05	0.15

TABLE VII.—ADDITIONS TO TABLE II, SPECIFICATIONS B 210 - 57 T.

Alloy		Temper	Wall Thickness, in.	Tensile Strength, psi		Yield Strength, min, psi	Elongation in 2 in. or 4 × Diameter, min, per cent	
ASTM Designation	AA Designation			Max	Min		Full-Section Specimen	Cut-out Specimen
GR20B	5652	O[b]	0.010 to 0.450	35 000	25 000	10 000[c]	...	...
		H34	0.010 to 0.450	...	34 000	26 000[c]	...	...
		H38	0.010 to 0.450	...	39 000	31 000[c]	...	...
GR40B	5254	O[b]	0.010 to 0.450	...	30 000	11 000[c]	...	...
		H34	0.010 to 0.450	...	39 000	29 000[c]	...	...
		H38	0.010 to 0.250	...	45 000	34 000[c]	...	...

ing to the ASTM Designation, as shown in the accompanying Table IV.

Section 7.—Revise the second sentence to add alloys GM31A, GR20B, and GR40B.

Table II.—Revise the Alloy Headings to add the AA designation corresponding to the ASTM designation, as shown in Table I.

Add tensile requirements for alloys GM31A, GR20B, and GR40B, as shown in the accompanying Table V.

Section 9.—Revise the first sentence to add alloys GM31A, GR20B, and GR40B following alloys G1B, GR20A, and GR40A, respectively.

Table IV.—Revise the title to add alloys GM31A, GR20B, and GR40B.

TABLE VIII.—ADDITIONS TO TABLE I, SPECIFICATION B 211 - 57 T.

Alloy		Aluminum, per cent	Copper, per cent	Iron, per cent	Silicon per cent	Manganese, per cent	Magnesium, per cent	Zinc, per cent	Chromium, per cent	Titanium, per cent	Lead, per cent	Bismuth, per cent	Other Elements	
ASTM Designation	AA Designation												Each	Total
Clad GM50 Core......	Alclad 5056	remainder	0.10	0.40	0.30	0.05 to 0.20	4.5 to 5.6	0.10	0.05 to 0.20	...	...	...	0.05	0.15
Cladding..	6253	remainder	0.10	0.50	[e]	...	1.0 to 1.5	1.6 to 2.4	0.15 to 0.35	...	...	...	0.05	0.15
GR20B.....	5652	remainder	0.04	[c]	[c]	0.01	2.2 to 2.8	0.20	0.15 to 0.35	...	...	...	0.05	0.15

[e] 45 to 65 per cent of magnesium content.

TABLE IX.—ADDITIONS TO TABLE II, SPECIFICATION B 211 - 57 T.

Alloy		Temper	Diameter or Thickness, in.	Tensile Strength, psi		Yield Strength, psi[a]	Elongation in 2 in. or 4 × Diameter, min, per cent[c]
ASTM	AA			Minimum	Maximum		
Clad GM50A......	Alclad 5056..	H38	0.120 to 0.192	54 000	...	47 000	...
GR20B..........	5652...	O	0.124 and under	...	32 000	...	...
			0.125 and over	25 000	32 000	9 500 min[b]	25
		H34	0.374 and under	34 000	...	26 000 min[b]	...
		H38	0.374 and under	39 000	...	...	...
		F	all	[d]	[d]	...	...

Appendix.—Delete the entire Appendix.

Tentative Specification for Aluminum Alloy Drawn Seamless Tubes (B 210 - 57 T):

Table I.—Add a column presenting the AA alloy designation corresponding to the ASTM alloy designation, shown in Table I; also add a Footnote *b* referenced to the column heading "AA Designation," renumbering subsequent footnotes accordingly, to read "[b] These AA alloy designations of the Aluminum Association were established in accordance with the American Standard Alloy Designation System for Wrought Aluminum (ASA No. H35.1—1957)."

Add chemical requirements for alloys GR20B and GR40B; also add the AA alloy designation to the ASTM designation, as shown in the accompanying Table VI.

TABLE X.—ADDITIONS TO TABLE I, SPECIFICATION B 221 - 57 T.

Alloy		Aluminum, per cent	Copper, per cent	Iron cent	Silicon, per cent	Manganese, per cent	Magnesium, per cent	Zinc, per cent	Chromium, per cent	Titanium, per cent	Beryllium, per cent	Other Elements	
ASTM Designation	AA Designation											Each	Total
GM31A...	5454..	remainder	0.10	[e]	[e]	0.50 to 1.0	2.4 to 3.0	0.25	0.05 to 0.20	0.20	...	0.05	0.15
GM40A...	5086..	remainder	0.10	0.50	0.40	0.20 to 0.7	3.5 to 4.5	0.25	0.05 to 0.25	0.15	...	0.05	0.15
GM41A...	5083..	remainder	0.10	0.40	0.40	0.30 to 1.0	4.0 to 4.9	0.25	0.05 to 0.25	0.15	...	0.05	0.15
GM51A...	5456..	remainder	0.20	[e]	[e]	0.50 to 1.0	4.7 to 5.5	0.25	0.05 to 0.20	0.20	0.0005	0.05	0.15
GR40B...	5254..	remainder	0.05	[f]	[f]	0.01	3.1 to 3.9	0.20	0.15 to 0.35	0.05	...	0.05	0.15

TABLE XI.—ADDITIONS TO TABLE II, SPECIFICATIONS B 221 - 57 T.

Alloy		Temper	Thickness, in.	Area, sq in.	Tensile Strength, psi		Yield Strength, min, psi	Elongation in 2 in. or 4 × Diameter, min, per cent
ASTM	AA				Min	Max		
GM31A...	5454....	O	5.000 and under	32 and under	31 000	41 000	12 000	14
		H112	5.000 and under	32 and under	31 000	...	12 000	12
		H311	5.000 and under	32 and under	31 000	...	16 000	8
GM40A...	5086.....	H112	all	all	35 000	...	18 000	12
GM41A...	5083....	O	all	all	38 000	...	16 000	16
		H112	all	all	40 000	...	24 000	12
GR40B....	5254....	O	all	...	30 000	41 000	11 000[g]	...
		H112	all	...	30 000	...	11 000[g]	...

Table II.—Add a column presenting the AA alloy designation corresponding to the ASTM alloy designation, as shown in Table I, adding also alloys GR20B and GR40B:

Add a new Footnote *f* referenced to the column heading "Elongation in 2 in. or 4 × Diameter, min, per cent," to read "[f] Elongation of full-section and cut-out sheet-type specimens is measured in 2 in.; of cut-out round specimens, in 4 × specimen diameter."

Add tensile requirements for alloys GR20B and GR40B; also add AA alloy designations corresponding to the ASTM alloys, as shown in the accompanying Table VII.

Section 7.—Revise the second sentence to add alloys GR20B and GR40B.

Table III.—Revise both the second and third column headings to add alloys GR20B and GR40B.

Table IV.—Revise both the second and third column headings to add alloys GR20B and GR40B.

Appendix.—Delete the entire Appendix.

Tentative Specification for Aluminum-Alloy Bars, Rods, and Wire (B 211 - 57 T):

Footnote 2.—Revise the last paragraph of Footnote *2* to read: "The 1957 revision included also the incorporation of the appropriate requirements of the Tentative Specifications for Aluminum Bars, Rods, and Shapes for Pressure Vessels (B 273 - 56 T). Specifications B 273 have accordingly been discontinued."

Table I.—Add a column presenting the AA alloy designation corresponding to the ASTM alloy designation as shown in Table I; also add a new Footnote *b* referenced to the column heading "AA Designation," renumbering subsequent footnotes accordingly, to read: "[b] These AA alloy designations of the Aluminum Association were established in accordance with the American Standard Alloy Designation System for Wrought Aluminum (ASA No. H35.1—1957)."

Add chemical requirements for alloys clad GM50A and GR20B; also add the AA alloy designation corresponding to the ASTM alloy designation, as shown in the accompanying Table VIII.

Table II.—Add a column presenting the AA alloy designation corresponding to the ASTM alloy designation, as shown in Table I.

Add tensile requirements for alloys clad GM50A and GR20B; also add AA alloy designation corresponding to ASTM alloy designation, as shown in the accompanying Table IX.

Section 7.—Revise the second sentence to add alloy GR20B.

New Section.—Add a new Section 13 entitled "Cladding Thickness," to read as follows, renumbering subsequent sections accordingly:

> 13. (*a*) The aluminum alloy coating of clad GM50A wire shall have a thickness of approximately 20 per cent of the total cross-sectional area of the wire.
>
> (*b*) When the thickness of the coating is to be determined on finished wire, transverse cross-sections of at least three wires from the lot shall be polished for microscopic examination. Using a magnification of 100 diameters, the coating thickness at four points, 90 deg apart, in each sample shall be measured and the average of the 12 measurements shall be taken as the thickness.

Appendix.—Delete the entire Appendix.

Tentative Specification for Aluminum Alloy Extruded Bars, Rods, and Shapes (B 221 - 57 T):

Table I.—Add a column presenting the AA alloy designation corresponding to the ASTM alloy designation, shown in Table I; also add a new Footnote *b*, referenced to the column heading "AA Designation," renumbering subsequent footnotes accordingly, to read "[b] These AA alloy designations of the Aluminum Association were established in accordance with the American Standard Alloy Designation System for Wrought Aluminum (ASA No. H35.1—1957)."

Change chemical requirements for alloy GM51A and add chemical requirements for alloys GM31A, GM40A, GM41A, and GR40B; also add the AA alloy designation corresponding to the ASTM alloy designation, as shown in the accompanying Table X.

Table II.—Add a column presenting the AA alloy designation corresponding to the ASTM alloy designation, as shown in Table I.

Add tensile requirements for alloys GM31A, GM40A, GM41A, and GR40B; also add AA alloy designation, as shown in the accompanying Table XI.

Section 7.—Revise the second sentence to add alloy GM31A, GM40A, GM41A, and GR40B.

Appendix.—Delete the entire Appendix.

Tentative Specification for Aluminum Alloy Drawn Seamless Tubes for Condensers and Heat Exchangers (B 234 - 57 T):

Table I.—Add a column presenting the AA alloy designation corresponding to the ASTM alloy designation as shown in Table I; also add a new Footnote *b* referenced to the column heading "AA Designation," renumbering subsequent footnotes accordingly, to read "[b] These AA alloy designations of the Aluminum

TABLE XII.—ADDITIONS TO TABLE I, SPECIFICATION B 235 – 57 T.

Alloy		Aluminum, per cent	Copper, per cent	Iron, per cent	Silicon, per cent	Manganese, per cent	Magnesium, per cent	Zinc, per cent	Chromium, per cent	Titanium, per cent	Other Elements, per cent	
ASTM Designation[a]	AA Designation[b]										Each	Total
GM31A...	5454...	remainder	0.10	[f]	[f]	0.50 to 1.0	2.4 to 3.0	0.25	0.05 to 0.20	0.20	0.05	0.15
GM51A[g]..	5456...	remainder	0.20	[f]	[f]	0.50 to 1.0	4.7 to 5.5	0.25	0.05 to 0.20	0.20	0.05	0.15
GR20B....	5652...	remainder	0.04	[d]	[d]	0.01	2.2 to 2.8	0.20	0.15 to 0.35	...	0.05	0.15
GR40B....	5254...	remainder	0.05	[d]	[d]	0.01	3.1 to 3.9	0.20	0.15 to 0.35	0.05	0.05	0.15

[g] For alloy GM51A the maximum beryllium content shall be 0.0005 per cent.

TABLE XIII.—ADDITIONS TO TABLE II, SPECIFICATION B 235 – 57 T.

Alloy		Temper	Thickness, in.	Area, sq in.	Tensile Strength, psi		Yield Strength, min, psi	Elongation in 2 in. or 4 × Diameter, min, per cent
ASTM Designation	AA Designation				Min	Max		
GM31A...	5454..	O	5.000 and under	32 and under	31 000	41 000	12 000	14
		H112	5.000 and under	32 and under	31 000	...	12 000	12
		H311	5.000 and under	32 and under	31 000	...	16 000	8
GM51A...	5456..	O	all	...	42 000	53 000	19 000	16
		H112	all	...	42 000	...	19 000	12
GR20B....	5652..	O	all	...	25 000	35 000	10 000[b]	...
		F	all	...	[f]	[f]	[f]	[f]
GR40B....	5254..	O	all	...	30 000	...	11 000[b]	...
		F	all	...	[f]	[f]	[f]	[f]

TABLE XIV.—ADDITIONS TO TABLE I, SPECIFICATIONS B 241 – 57 T.

ASTM Designation[a]	Alloy GM51A	Alloy GM31A
AA Designation[b]	Alloy 5456	Alloy 5454
Aluminum, per cent	remainder	remainder
Copper, per cent	0.20	0.10
Iron, per cent	[c]	[c]
Silicon, per cent	[c]	[c]
Manganese, per cent	0.50 to 1.0	0.50 to 1.0
Magnesium, per cent	4.7 to 5.5	2.4 to 3.0
Zinc, per cent	0.25	0.25
Chromium, per cent	0.05 to 0.20	0.05 to 0.20
Titanium, per cent	0.20	0.20
Beryllium, per cent	0.0005	...
Other Elements:		
Each, per cent	0.05	0.05
Total, per cent	0.15	0.15

[c] Iron plus silicon shall not exceed 0.40 per cent.

Association were established in accordance with the American Standard Alloy Designation System for Wrought Aluminum (ASA No. H35.1—1957)."

Table II.—Add a column presenting the AA alloy designation corresponding to the ASTM alloy designation, as shown in Table I.

Add a new Footnote *b* referenced to the column heading "Elongation in 2 in. or 4 × Diameter, min, per cent," to read: "[b] Elongation of full-section and cut-out sheet-type specimens is measured in 2 in.; of cut-out round specimens, in 4 × specimen diameter."

Section 13.—Change the first sentence to read: "The tubing shall be capable of being flattened along any element under a gradually applied load until the *maximum outside diameter in the line of pressure* is not more than eight times the wall thicknesss."

Appendix II.—Delete the entire Appendix II.

Tentative Specification for Aluminum Alloy Extruded Tubes (B 235 - 57 T):

Table I.—Add a column presenting the AA alloy designation corresponding to the ASTM alloy designation, as shown in Table I; also add a new footnote *b* referenced to the column heading "AA Designation," renumbering subsequent footnotes accordingly, to read: "[b] These AA alloy designations of the Aluminum Association were established in accordance with the American Standard Alloy Designation System for Wrought Aluminum (ASA No. H35.1—1957)."

Add chemical requirements for alloys GM31A, GM51A, GR20B, and GR40B; also add the AA alloy designation corresponding to the ASTM alloy designation, as shown in the accompanying Table XII.

Section 7.—Revise the second sentence to add alloys GM31A, GM51A, GR20B, and GR40B.

Table II.—Add a column presenting the AA alloy designation corresponding to the ASTM alloy designation, as shown in Table I.

Add a new Footnote *j* referenced to the column heading "Elongation in 2 in. or 4 × Diameter, min. per cent," to read: "[j] Elongation of full-section and cut-out sheet-type specimens is measured in 2 in.; of cut-out round specimens, in 4 × specimen diameter.

Add tensile requirements for alloys GM31A, GM51A, GR20B, and GR40B; also add AA alloy designations corresponding to the ASTM alloy designation, as shown in the accompanying Table XIII.

Appendix.—Delete the entire Appendix.

Tentative Specification for Aluminum Bars for Electrical Purposes (B 236 - 57 T):

Section 6.—Change the value for resistivity from "0.07640" to "0.0764 ohm—gram/meter^2 and for conductivity from "61" to "61.0 per cent."

Section 9 (c).—Revise to read as follows:

> (*c*) Electrical resistivity or conductivity shall be determined in accordance with the Standard Method of Test for Resistivity of Electrical Conductor Materials (ASTM Designation: B 193) or the Eddy Current Method, provided that in case of dispute methods in accordance with Method B 193 shall be the basis for acceptance.

Tentative Specification for Aluminum Alloy Pipe (B 241 - 57 T):

Table I.—Revise the column headings to present the AA alloy designation corresponding to the ASTM alloy designation; add new Footnotes *a* and *b* referenced to column 1, renumbering subsequent footnotes accordingly, as follows:

ASTM Designation[a]	Alloy GS10A	Alloy GS11A	Alloy GS11C	Alloy M1A
AA Designation[b]	Alloy 6063	Alloy 6061	Alloy 6062	Alloy 3003

[a] These alloy designations were established in accordance with the Recommended Practice for Codification of Light Metals and Alloys, Cast and Wrought (ASTM Designation: B 275).

[b] These alloy designations of the Aluminum Association were established in accordance with the American Standard Alloy Designation System for Wrought Aluminum (ASA No. H35.1—1957).

Add chemical requirements for alloys GM31A and GM51A, a new footnote, and the AA alloy designation corresponding to the ASTM designation, as shown in the accompanying Table XIV.

Section 7.—Revise the second sentence to add alloys GM31A and GM51A.

Table II.—Add a column presenting the AA alloy designation corresponding to the ASTM alloy, as shown in Table I; also delete Footnote *b* and its reference in column titled "Alloy," renumbering subsequent footnotes accordingly.

Add a new Footnote *d* referenced to the column heading "Elongation in 2 in. or 4 × Diameter, min, per cent," to read: "[d] Elongation of full-section and cut-out sheet-type specimens is measured in 2 in.; of cut-out round specimens, in 4 × specimen diameter."

Add tensile requirements for alloys GM31A and GM51A; also add AA alloy designation corresponding to ASTM alloy, as follows:

Alloy		Temper	Pipe Size, in.	Tensile Strength, min, psi	Yield Strength, min, psi[b]	Elongation in 2 in. or 4 × Diameter, min, per cent[d]
ASTM Designation	AA Designation					
GM31A.........	5454......	O	1 and over	31 000	12 000	12
		H112	1 and over	31 000	12 000	10
GM51A..........	5456......	O	All	42 000	19 000	16
		H112	All	42 000	19 000	12

Appendix.—Delete the entire Appendix.

Tentative Specification for Aluminum Alloy Die Forgings (B 247 - 57 T):

Table I.—Add a column presenting the AA alloy designation corresponding to the ASTM alloy designation as shown in Table I; add a new footnote *b*, referenced to the column "AA Designation," renumbering subsequent footnotes accordingly, to read: "[b] These AA alloy designations of the Aluminum Association were established in accordance with the American Standard Alloy Designation System for Wrought Aluminum (ASA No. H35.1—1957)."

Table II.—Add a column presenting the AA alloy designation corresponding to the ASTM alloy designation, as shown in Table I. Revise the temper designation for alloys 990A and M1A from the present "F" to read "H112."

Appendix.—Delete the entire Appendix.

Tentative Specification for Aluminum Alloy Drawn Seamless Coiled Tubes for Special Purpose Applications (B 307 - 57 T):

Table I.—Add a column presenting the AA alloy designation corresponding to the ASTM alloy designation, as shown in Table I; also add a Footnote *e* referenced to the column heading "AA Designation," to read: "[e] These AA alloy designations of the Aluminum Association were established in accordance with the American Standard Alloy Designation System for Wrought Aluminum (ASA No. H35.1—1957)."

Table II.—Add a column presenting the AA alloy designation corresponding to the ASTM alloy designation, as shown in Table I.

Section 13 (b).—Change the first sentence to read: "As an alternate to the expansion test, the tube shall be capable of being flattened along any element under a gradually applied load until the *maximum outside diameter in the line of pressure* is not more than three times the wall thickness."

Appendix.—Delete the entire Appendix.

Tentative Specification for Aluminum Alloy Standard Structural Shapes, Rolled or Extruded (B 308 - 57 T):

Table I.—Add a column presenting the AA alloy designation corresponding to the ASTM alloy designation, as shown in Table I; also add a new Footnote *b*, referenced to the column heading "AA Designation," renumbering subsequent footnotes accordingly, to read: "[b] These AA alloy designations of the Aluminum Association were established in accordance with the American Standard Alloy Designation System for Wrought Aluminum (ASA No. H35.1—1957)."

Change the chemical requirements for alloy GM51A and add chemical requirements for alloys GM31A, GM40A, and GM41A; also add the AA alloy designation corresponding to the ASTM alloy designation, as shown in the accompanying Table XV.

TABLE XV.—ADDITIONS TO TABLE I, SPCIFICATION B 308 - 57 T.

Alloy		Aluminum, per cent	Cooper, per cent	Iron, per cent	Silicon, per cent	Manganese, per cent	Magnesium, per cent	Zinc, per cent	Chromium, per cent	Titanium, per cent	Beryllium, per cent	Others Element	
ASTM Designation[a]	AA Designation[b]											Each	Total
GM31A...	5454..	remainder	0.10	[b]	[b]	0.50 to 1.0	2.4 to 3.0	0.25	0.05 to 0.20	0.20	...	0.05	0.15
GM40A...	5086..	remainder	0.10	0.50	0.40	0.20 to 0.7	3.5 to 4.5	0.25	0.05 to 0.25	0.15	...	0.05	0.15
GM41A..	5083..	remainder	0.10	0.40	0.40	0.30 to 1.0	4.0 to 4.9	0.25	0.05 to 0.25	0.15	...	0.05	0.15
GM51A...	5456..	remainder	0.20	[b]	[b]	0.50 to 1.0	4.7 to 5.5	0.25	0.05 to 0.20	0.20	0.0005	0.05	0.15

TABLE XVI.—ADDITIONS TO TABLE II, SPECIFICATION B 308 - 57 T.

Alloy		Temper	Thickness, in.	Area, sq in.	Tensile Strength, psi		Yield Strength, min, psi	Elongation in 2 in. or 4 × diameter, min, per cent
ASTM Designation	AA Designation				Min	Max		
GM31A....	5454..	O	5.000 and under	32 and under	31 000	41 000	12 000	14
		H112	5.000 and under	32 and under	31 000	...	12 000	12
		H311	5.000 and under	32 and under	31 000	...	16 000	8
GM40A....	5086...	H112	all	all	35 000	...	18 000	12
GM41A....	5083..	O	all	all	38 000	...	16 000	16
		H112	all	all	40 000	...	24 000	12

Table II.—Add a column presenting the AA alloy designation corresponding to the ASTM alloy designation, as shown in Table I; also delete Footnote *a*, renumbering subsequent footnotes accordingly.

Add tensile requirements for alloys GM31A, GM40A, and GM41A; also add AA alloy designation corresponding to the ASTM alloy designation, as shown in the accompanying Table XVI.

Add requirements for alloy GS11C-T62 reading identically to those now included in Table II for alloy GS11A-T62.

Appendix.—Delete the entire Appendix.

Tentative Specification for Aluminum Alloy Round Welded Tubes (B 313 - 57 T):

Table I.—Add a column presenting the AA alloy designation corresponding

to the ASTM alloy designation, as shown in Table I; also add a new Footnote *b* referenced to the column heading "AA Designation," renumbering subsequent footnotes accordingly, to read: "[b] These AA alloy designations of the Aluminum Association were established in accordance with the American Standard Designation System for Wrought Aluminum (ASA No. H35.1—1957)."

Table II.—Add a column presenting the AA alloy designations corresponding to the ASTM alloy designation, as shown in Table I.

Appendix.—Delete the entire Appendix.

Tentative Specification for Aluminum Alloy Rivet and Cold Heading Wire and Rods (B 316 - 57 T):

Table I.—Add a column presenting the AA alloy designation corresponding to the ASTM alloy designation, as shown in Table I; also add a new Footnote *b* referenced to the column heading "AA Designation," renumbering subsequent footnotes accordingly, to read: "[b] These AA alloy designations of the Aluminum Association were established in accordance with the American Standard Alloy Designation System for Wrought Aluminum (ASA No. H35.1—1957)."

Table II.—Add a column presenting the AA alloy designation corresponding to the ASTM alloy designation, as shown in Table I.

Table III.—Add a column presenting the AA alloy designation corresponding to the ASTM alloy designation; also establish a separate column for temper designation, as follows:

Alloy		Temper
ASTM Designation	AA Designation	
CG30A...........	2117.......	T4
CG42A...........	2024.......	T4
CM41A..........	2017.......	T4
GS11A...........	6061.......	T6
GS11B...........	6053.......	T61
ZG62A...........	7075.......	T6

Appendix.—Delete the entire Appendix.

Tentative Specification for Extruded Aluminum Alloy Bars, Rods, Pipe, and Structural Shapes for Electrical Purposes (B 317 - 57 T):

Section 1.—Change to read as follows:

1. This specification covers ASTM alloy GS10B[3] (AA alloy 6101)[4] bar, rod, pipe, and structural shapes in tempers T6, T61, and T62 for use as electrical conductors.

New Footnotes.—Add new Footnotes 3 and 4 to read as follows:

[3] This ASTM alloy designation was established in accordance with the Recommended Practice for Codification of Light Metals and Alloys, Cast and Wrought (ASTM Designation: B 275).

[4] This AA alloy designation of the Aluminum Association was established in accordance with the American Standard Alloy Designation System for Wrought Aluminum (ASA No. H35.1—1957).

Appendix II.—Delete Appendix II.

Tentative Specification for Type A and Type B Aluminum Alloy Drawn Annealed Seamless Coiled Tubes (B 318 - 57 T):

Table II.—Revise to add a column presenting the AA alloy designation corresponding to the ASTM alloy designation, as shown in Table I; also add a new Footnote *b* referenced to the column heading "AA Designation," renumbering subsequent footnotes accordingly, to read: "[b] These AA alloy designations of the Aluminum Association were established in accordance with the American Standard Alloy Designation System for Wrought Aluminum (ASA No. H35.1—1957)."

Table III.—Revise to add a column presenting the AA alloy designation corresponding to the ASTM alloy designation, shown in Table I.

Appendix.—Delete the entire Appendix.

ADOPTION OF TENTATIVE AS STANDARD WITHOUT REVISION

The committee recommends that the

Tentative Specification for Magnesium-Base Alloy Sheet and Plate (**B 90 – 57 T**) be approved for reference to letter ballot of the Society for adoption as standard without revision.

ADOPTION OF TENTATIVES AS STANDARD WITH REVISION

The committee recommends that the following tentatives be approved for reference to letter ballot of the Society for adoption as standard with revisions as indicated:

TABLE XVII.—ADDITIONS TO TABLE II, SPECIFICATION B 80 – 57 T.

Alloy	Condition	Yield Strength (0.2 per cent offset), min, psi
AZ63A	-F (as-cast)	10 000
AZ63A	-T4 (solution heat-treated)	11 000
AZ81A	-T4 (solution heat-treated)	10 000
AZ91C	-F (as-cast)	10 000
AZ91C	-T4 (solution heat-treated)	11 000
AZ92A	-F (as-cast)	10 000
AZ92A	-T4 (solution heat-treated)	11 000

Tentative Specification for Magnesium-Base Alloy Sand Castings (B 80 – 57 T):

Table II.—Add to Table II the minimum tensile yield strengths as shown in the accompanying Table XVII.

Table III.—Revise the values given for the following alloys to read as shown in the accompanying Table XVIII.

New Table.—Add a new Table V to read as shown in the accompanying Table XIX.

New Explanatory Note.—Add a new Explanatory Note 5 to read as follows:

NOTE 5.—The minimum properties of bars cut from castings shown in Table V are given for information only and do not form a part of this specification. The minimums in Table V are available for most castings although better properties may be obtained in some castings or selected sections of other castings. All properties of bars cut from castings require special agreement between the purchaser and manufacturer.

Tentative Specification for Magnesium-Base Alloy Forgings (B 91 – 57 T):

Table I.—In Table I change the maximum silicon percentages on alloys AZ31B, AZ61A, and AZ80A from "0.30" to "0.10." Add a maximum of 0.04 per cent calcium on alloy AZ31B.

Tentative Specification for Magnesium-Base Alloys in Ingot Form for Sand Castings, Die Castings, and Permanent Mold Castings (B 93 – 57 T):

Table I.—Delete alloys AM80A, AM100B and M1B from Table I. Add alloy AZ81A to Table I with the following composition:

Aluminum, per cent	7.2–8.0
Manganese, min, per cent	0.15
Zinc, per cent	0.50–0.9
Silicon, max, per cent	0.20
Copper, max, per cent	0.08
Nickel, max, per cent	0.01
Other impurities, max, per cent	0.30
Magnesium	remainder

Tentative Specification for Magnesium-Base Alloy Bars, Rods, and Shapes (B 107 – 57 T):

Table I.—Lower the silicon content of alloys AZ31B, AZ61A, AZ80A, and M1A from 0.30 per cent max to 0.10 per cent max. Add a maximum of 0.04 per cent on calcium for alloy AZ31B in the same table.

Tentative Specification for Magnesium-Base Alloy Permanent Mold Castings (B 199 – 57 T):

Table II.—Add the following minimum tensile yield strengths to Table II:

Alloy	Condition	Yield Strength (0.2 per cent offset), min, psi
AZ91C	-F (as-cast)	10 000
	-T4 (solution heat-treated)	11 000
AZ92A	-F (as-cast)	10 000
	-T4 (solution heat-treated)	11 000

TABLE XVIII.—REVISIONS FOR TABLE III, SPECIFICATION B 80 - 57 T.

Alloy	Condition	Typical or Minimum	Yield Strength (0.2 per cent offset), min, psi	Unit Deformation, inch per inch of gage length
AZ63A	-F (as-cast)	minimum	10 000	0.0035
	-T5 (artificially aged only)	minimum	11 000	0.0037
	-T4 (solution heat-treated)	minimum	11 000	0.0037
AZ81A	-T4 (solution heat-treated)	minimum	10 000	0.0035
AZ91C	-F (as-cast)	minimum	10 000	0.0035
	-T4 (solution heat-treated)	minimum	11 000	0.0037
AZ92A	-F (as-cast)	...	10 000	0.0035
	-T5 (artificially aged only)	...	11 000	0.0037
	-T4 (solution heat-treated)	...	11 000	0.0037

TABLE XIX.—TENSION TEST MINIMUMS FOR SPECIMENS CUT FROM CASTINGS.
(New Table V, Specification B 80 - 57 T.)

Alloy and Temper	Tensile Strength		Yield Strength (0.2 per cent offset)		Elongation in 2 in., Average[a]
	Average, psi	Minimum, psi	Average, psi	Minimum, psi	
AZ63A					
T4	25 500	17 000	10 000	9 000	...
T6	25 500	17 000	14 500	12 000	...
AZ81A					
T4	25 500	17 000	10 000	9 000	...
AZ91C					
T4	25 500	17 000	10 000	9 000	...
T6	25 500	17 000	14 500	12 000	...
AZ92A					
T4	25 500	17 000	10 000	9 000	...
T6	25 500	17 000	16 000	13 500	...
EZ33A					
T5 (room temperature)	15 000	13 000	12 500	11 000	...
T5 (500 F)	...	10 000	...	6 000	...
HK31A					
T6 (room temperature)	23 000	19 000	11 700	10 500	...
T6 (500 F)	...	10 000	...	6 000	...
HZ32A					
T5 (room temperature)	23 000	19 000	11 700	10 500	...
T5 (500 F)	...	10 000	...	6 000	
ZK51A					
T5	29 000	24 000	17 000	14 000	...
ZH62A					
T5	31 500	26 500	17 500	15 500	...

[a] The average per cent elongation of specimens cut from castings shall be not less than 25 per cent of the values specified for separately cast test bars.

Note.—The above values, both average and minimum, are representative of the weakest areas in any type casting for the composition listed, that is, adjacent to sprues and risers. Higher design properties for designated areas in castings are normally obtained and shall be as specified in the order.

Table III.—Add the following new conditions to Table III:

Alloy	Condition	Yield Strength (0.2 per cent offset), min, psi	Unit Deformation, inch per inch of gage length
AZ91C....	-F (as-cast)......	10 000	0.0035
	-T4 (solution heat-treated).....	11 000	0.0037
AZ92A....	-F (as-cast)......	10 000	0.0035
	-T4 (solution heat-treated).....	11 000	0.0037

Tentative Specification for Magnesium-Base Alloy Extruded Tubes (B 217 – 57 T):

Table I.—Lower the maximum silicon permitted in Table I on alloys AZ31B, AZ61A, and M1A from 0.30 per cent to 0.10 per cent. Add a maximum of 0.04 per cent on calcium in alloy AZ31B in the same table.

REVISIONS OF STANDARDS, IMMEDIATE ADOPTION

The committee recommends for immediate adoption revisions as indicated of the following standards, and accordingly asks for the necessary nine-tenths affirmative vote at the Annual Meeting in order that the revisions may be referred to letter ballot of the Society:

Standard Specification for Aluminum-Base Alloys in Ingot Form for Sand Castings, Die Castings, and Permanent Mold Castings (B 179 – 57):

Table I.—Delete alloy SC54A-B. Add alloy GM70B with the chemical requirements and a new Footnote *e* as shown in the accompanying Table XX.

New Section.—Add a new Section 2 entitled "Basis of Purchase," to read as follows, renumbering the subsequent sections accordingly:

2. Orders for material under this specification shall include the following information:

(*1*) Quantity in pieces or pounds,

TABLE XX.—ADDITIONS TO TABLE I, SPECIFICATION B 179 – 57.

Aluminum, per cent...........	remainder
Copper, per cent..............	0.05
Iron, per cent................	0.20[e]
Silicon, per cent.............	0.15[e]
Manganese, per cent...........	0.10 to 0.25
Magnesium, per cent..........	6.3 to 7.5
Zinc, per cent................	...
Chromium, per cent............	...
Titanium, per cent............	0.10 to 0.25
Other elements, each, per cent...	0.05
Other elements, total, per cent...	0.15

[e] The total content of iron plus silicon shall not exceed 0.30 per cent.

TABLE XXI.—COLOR CODING OF ALUMINUM-BASE INGOTS.
(New Table II, Specification B 179 – 57)

Alloy	Color	Alloy	Color
CG 100A	Red & White	SC 51A	Green
CN 42A	Red & Orange	SC 64A	Brown & Yellow
C 4A	Red & Brown	SC 64B	Yellow
CS 42A	Red & Yellow	SC 64C	Brown & Black
CS 43A	Red	SC 82A	Brown & Green
CS 66A	Red & Green	SC 84A-B	Brown
CS 72A	Red & Black	SC 103A	Brown & White
CS 104A	Red & Purple	SC 114A	Orange
		SC 122A	Brown & Blue
G 4A	Blue & White	SG 70A	Purple
G 8A	Blue & Red	SG 100A-B	Purple & White
G 10A	Blue	SN 122A	Orange & Green
GM 70B	Blue & Orange		
GS 42A	Blue & Yellow	ZC 60A	Purple & Orange
GZ 42A	Blue & Green	ZC 81A-B	Purple & Yellow
		ZG 32A	Green & Yellow
S 5A	Black & Yellow	ZG 42A	Purple & Green
S 5B	Black	ZG 61A	Orange & Yellow
S 5C	Black & Green	ZG 61B	Orange & Black
S 12A-B	Black & White		

TABLE XXII.—COLOR CODING OF ALUMINUM-BASE INGOTS.
(New Table III; Specification B 179 – 57)

	White	Black	Red	Yellow	Blue	Green	Brown	Purple	Orange
White		S12A-B	CG100A		G4A		SC103A	SG100A-B	
Black	S12A-B	**S5B**	CS72A	S5A		S5C	SC64C		ZG61B
Red	CG100A	CS72A	**CS43A**	CS42A	G8A	CS66A	C4A	CS104A	CN42A
Yellow		S5A	CS42A	**SC64B**	GS42A	ZG32A	SC64A	ZC81A-B	ZG61A
Blue	G4A		G8A	GS42A	**G10A**	GZ42A	SC122A		GM70B
Green		S5C	CS66A	ZG32A	GZ42A	**SC51A**	SC82A	ZG42A	SN122A
Brown	SC103A	SC64C	C4A	SC64A	SC122A	SC82A	**SC84A-B**		
Purple	SG100A-B		CS104A	ZC81A-B		ZG42A		**SG70A**	ZC60A
Orange		ZG61B	CN42A	ZG61A	GM70B	SN122A		ZC60A	**SC114A**

(*2*) Alloy (Section 5, Table I),

(*3*) Form; by agreement between the purchaser and vendor the approximate form and weight of each piece or ingot may be specified.

(*4*) Whether marking for identification is required, including marking pattern if required (Section 9), and

(*5*) Place of inspection (Section 11).

New Section.—Add a new Section 9 entitled "Marking for Identification," to read as follows, renumbering the subsequent sections accordingly:

9. When color identification marking of ingots is specified on the order, ingots shall be color coded as shown in the Appendix. Color may be applied by spray can or brush. Color pattern on ingots may be either stripe or dot at the request of the purchaser. Either one side or two adjacent ingot sides may be marked at the request of the purchaser.

Appendix.—Add an Appendix containing the color code referred to in the new Section 9, including the new Tables II and III as shown in the accompanying Tables XXI and XXII:

APPENDIX

When color identification marking of ingots is specified in the order the following color code, as shown in Tables II and III, shall be used.

Standard Recommended Practice for Codification of Light Metals and Alloys, Cast and Wrought (B 275 – 55):

New Footnote.—Add the following Footnote 3 to Section 2(*a*) to read as follows:

[3] However, the Committee B-7 subcommittee having jurisdiction over this recommended practice may tentatively assign designations for alloys and unalloyed metals before their chemical composition limits are included in ASTM Specifications.

Table I.—Delete the word "Beryllium" following the letter "L," replacing it with "Lithium."

New Paragraph.—Add a new Paragraph 5 entitled "Granular Metal Products," to read as follows:

5. Designation of light metals and alloys formed as a wrought product from granular metal powders or granules shall be by use of the prefix (P) in parentheses to the letter or letters forming the alloy designation.

REAPPROVAL OF STANDARDS

The committee recommends the reapproval of the following standards which have stood for six or more years without revision:

Standard Method of Test for:

Dielectric Strength of Anodically Coated Aluminum (**B 110 – 45**),

Sealing of Anodically Coated Aluminum (**B 136 – 45**), and

Weight of Coating on Anodically Coated Aluminum (**B 137 – 45**).

APPENDIX II

ATMOSPHERIC EXPOSURE OF ALUMINUM AND MAGNESIUM SAND AND PERMANENT MOLD CASTINGS*

By L. H. Adam[1] and Marie Dougherty[1]

The Atmospheric Exposure Program of Light Metals and Alloys as outlined to the Society in 1953[2] is presently being conducted by Committee B-7. The scope of the program includes both wrought and cast material exposed at five test sites, representing the rural conditions of State College, Pa., the industrial conditions of New York City, and the marine conditions along the Atlantic coast at Kure Beach, N. C., along the Pacific coast at Point Reyes, Calif., as well as along the Gulf coast at Freeport, Tex. Previous reports were presented to the Society in 1954[3] and 1955,[4] and contained data on material exposed for a 6-month and 1-yr period.

This report pertains to results exposed on cast aluminum and magnesium alloys for 3 yr at the 5 test sites. Six cast tensile specimens of each alloy, were removed from the exposure racks after a 3-yr period, and a similar group of specimens retained under control room conditions in sealed containers for 3 yr, were tested for mechanical properties.

Tables I to XXV inclusive show the yield strength, tensile strength and per cent elongation in 2 in., recorded on six samples removed from each of the five exposure sites. The bar graphs in Figs. 1, 2, and 3 illustrate the average tensile strength and per cent elongation for each exposure site as compared to the average 3-yr control values. In general, the aluminum and magnesium alloys show a loss of strength no greater than 5 per cent. However, the effect of exposure on the ductility of the material indicates that the majority of the values change no more than 20 per cent after 3-yr exposure.

From time to time, as more exposed samples are tested, the data will be presented for your review. The excellent cooperation of the participating members of the Society is gratefully acknowledged. Their continued interest in this program in the future will help increase the knowledge we already have accumulated concerning the performance of these lightweight metals.

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* Presented at the Sixty-first Annual Meeting of the Society, June 22-27, 1958.

[1] Materials Engineer and Physical Science Aide, respectively, Frankford Arsenal, Pitman Dunn Laboratories, Philadelphia, Pa.

[2] L. H. Adam, "Atmospheric Exposure of Light Metals," Appendix to Report of Committee B-7 on Light Metals and Alloys, Cast and Wrought, *Proceedings*, Am. Soc. Testing Mats., Vol. 53, pp. 227-250 (1953).

[3] L. H. Adam, "Atmospheric Exposure of Aluminum and Magnesium Sand and Permanent Mold Casting," Appendix to Report of Committee B-7 on Light Metals and Alloys, Cast and Wrought, *Proceedings*, Am. Soc. Testing Mats., Vol. 54, pp. 270-294 (1954).

[4] L. H. Adam and M. Dougherty, "Atmospheric Exposure of Wrought Aluminum and Magnesium Alloys," Appendix to Report of Committee B-7 on Light Metals and Alloys, Cast and Wrought, *Proceedings*, Am. Soc. Testing Mats., Vol. 55, pp. 284-311 (1955).

TABLE I. - DATA ON ALUMINUM ALLOY NO. 55B-F, SANDCASTING.

ASTM Specification Requirements	3 YR. CONTROL	3-YEARS EXPOSURE STATE COLLEGE	NEW YORK	KURE BEACH	POINT REYES	FREE-PORT
YIELD STRENGTH, 0.2 PER CENT OFFSET, PSI						
7000 psi, min	12000	12000	11400	11000	11500	11700
	12100	11200	11700	11500	11200	11400
	12100	12100	12000	12100	12100	11900
	11900	11700	11400	11500	11600	11900
	12000	11500	11400	11100	10900	11600
	12100	11700	-	12400	11200	12400
AVERAGE	12000	11700	11600	11600	11400	11800
TENSILE STRENGTH, PSI						
17000 psi, min	23400	23500	22800	23100	21300	22900
	23300	22600	23100	23200	19800	22800
	23200	22500	22700	23400	21000	22000
	22600	22900	23400	22600	21000	22700
	23500	22400	22000	23100	21000	23900
	23200	23000	—	23000	21300	22400
AVERAGE	23200	22800	22800	23100	20900	22800
ELONGATION IN 2 IN., PER CENT						
3.0 per cent, min	4.1	3.4	4.2	3.6	2.4[a]	3.1
	4.1	3.6	3.8	4.3	2.2[a]	2.7[a]
	3.6	3.4	3.7	4.2	3.2	3.7
	3.5	3.5	3.6	3.6	3.4	3.5
	3.4	3.9	3.3	4.1	2.7[a]	3.6
	4.1	3.8	—	4.5	2.8[a]	2.8[a]
AVERAGE	3.8	3.6	3.7	4.1	2.8[a]	3.2

[a] LOW

NOTE. - ALL SPECIFICATIONS ON ALUMINUM ALLOY SANDCASTINGS INCLUDE TABLE II OF ASTM TENTATIVE SPECIFICATIONS B26-56T

TABLE II. - DATA ON ALUMINUM ALLOY NO. C4A-T6, SANDCASTING.

ASTM Specification Requirements	3 YR. CONTROL	3-YEARS EXPOSURE STATE COLLEGE	NEW YORK	KURE BEACH	POINT REYES	FREE-PORT
YIELD STRENGTH, 0.2 PER CENT OFFSET, PSI						
20000 psi, min	33500	32100	32000	32300	30400	32300
	33500	32500	32300	32300	30100	32000
	33700	32300	32500	31300	30800	31600
	33100	31700	32400	31100	30200	31500
	33000	32000	32200	31500	30800	32700
	33400	32200	31700	31500	30400	32700
AVERAGE	33400	32100	32200	31700	30500	32100
TENSILE STRENGTH, PSI						
32000 psi, min	44100	43900	40800	41000	37600	40900
	45200	44200	42300	41400	38900	41600
	45700	43700	43700	41800	38500	40100
	44800	44400	41500	39700	38600	40300
	44300	44600	43000	41100	39200	39800
	45200	43000	41000	40900	38100	39400
AVERAGE	44900	44000	42100	41000	38100	40400
ELONGATION IN 2 IN., PER CENT						
3.0 per cent, min	4.1	3.5	2.9[a]	3.1	2.2[a]	2.9[a]
	4.8	3.4	3.8	3.0	3.3	3.0
	4.6	4.2	4.3	3.2	2.5[a]	2.2[a]
	3.5	4.0	3.3	2.5[a]	2.6[a]	2.6[a]
	4.6	4.5	3.9	3.7	2.5[a]	2.2[a]
	4.9	3.8	2.7[a]	2.7[a]	2.6[a]	3.1
AVERAGE	4.4	3.9	3.5	3.0	2.6[a]	2.7[a]

[a] LOW

TABLE III. - DATA ON ALUMINUM ALLOY NO. SC51A-T6, SANDCASTING.

ASTM Specification Requirements	3 YR. CONTROL	3-YEARS EXPOSURE STATE COLLEGE	NEW YORK	KURE BEACH	POINT REYES	FREE-PORT
	YIELD STRENGTH, 0.2 PER CENT OFFSET, PSI					
20000 psi, min	30200	29600	30400	29900	27000	30900
	31200	29600	30300	33700	30300	30500
	31000	33800	30200	30900	30200	30700
	31100	29000	29500	29900	30700	30000
	31100	29200	30400	30300	29600	31500
	30500	29100	30400	34600	30400	30600
AVERAGE	30900	30100	30200	31600	30000	30700
	TENSILE STRENGTH, PSI					
32000 psi, min	36900	37300	37900	37800	33300	36500
	39500	37800	37100	39100	35100	36400
	38700	40400	38000	36800	35600	36700
	38100	36400	36200	36800	35600	35300
	38400	37000	34700	38500	36300	37100
	38000	36800	37800	40300	35900	37900
AVERAGE	38200	37600	36900	38200	35300	36700
	ELONGATION IN 2 IN, PER CENT					
2.0 per cent, min	2.6	2.7	2.4	2.1	0.7[a]	1.4[a]
	2.4	2.9	2.1	1.8[a]	1.6[a]	1.0[a]
	2.7	1.6[a]	2.5	1.6[a]	1.5[a]	1.4[a]
	2.4	2.6	2.2	1.7[a]	1.5[a]	1.3[a]
	2.4	2.2	1.9[a]	2.2	2.3	1.8[a]
	1.9[a]	2.3	2.3	1.9[a]	1.0[a]	1.4[a]
AVERAGE	2.4	2.4	2.2	1.9[a]	1.4[a]	1.5[a]

[a] Low

TABLE IV. - DATA ON ALUMINUM ALLOY NO. SG70A-T6, SANDCASTING.

ASTM Specification Requirements	3 YR. CONTROL	3-YEARS EXPOSURE STATE COLLEGE	NEW YORK	KURE BEACH	POINT REYES	FREE-PORT
	YIELD STRENGTH, 0.2 PER CENT OFFSET, PSI					
20000 psi, min	25900	24700	25800	25400	26500	25300
	27100	25200	26400	25400	27800	26500
	25400	29100	24600	27300	26500	25800
	24700	26500	26700	25600	25900	27000
	25700	24600	26000	25000	26200	24900
	26600	24400	26300	24000	25900	25600
AVERAGE	25900	25800	26100	25500	26500	25900
	TENSILE STRENGTH, PSI					
30000 psi, min	35000	34900	33900	35700	33800	34600
	35800	34000	35000	35300	34900	34800
	34800	36000	34500	36000	35300	35700
	33500	34500	34100	34600	33800	35700
	34400	35100	33800	34400	33600	33700
	35400	33900	34200	34000	35000	34200
AVERAGE	34800	34700	34200	34900	34400	34800
	ELONGATION IN 2 IN, PER CENT					
3.0 per cent, min.	4.4	4.6	4.0	4.7	3.5	3.8
	4.4	4.3	3.9	4.8	4.3	3.3
	4.8	5.6	4.7	3.7	4.6	4.2
	4.0	4.7	2.7[a]	3.9	3.5	3.7
	4.4	5.1	3.6	4.4	3.4	3.3
	4.3	4.1	4.0	4.0	5.0	3.5
AVERAGE	4.4	4.7	3.8	4.3	4.1	3.6

[a] Low

TABLE V. - DATA ON ALUMINUM ALLOY NO. SC64B-T6, SANDCASTING.

TABLE VI. - DATA ON ALUMINUM ALLOY NO. ZG61A-T5, SANDCASTING.

TABLE V. - DATA ON ALUMINUM ALLOY NO. SC64B-T6, SANDCASTING.

ASTM Specification Requirements	3 YR. CONTROL	3-YEARS EXPOSURE				
		STATE COLLEGE	NEW YORK	KURE BEACH	POINT REYES	FREE PORT
		YIELD STRENGTH, 0.2 PER CENT OFFSET, PSI				
20000 PSI, min	33500	34100	34300	35000	33600	34600
	35000	34600	33600	33600	34600	34600
	35800	33800	33900	33800	34300	34800
	35200	34400	34600	34500	33600	35200
	35700	34100	34300	34800	32000	34100
	35400	34700	34800	33800	33400	34500
AVERAGE	35400	34300	34300	34300	33600	34600
		TENSILE STRENGTH, PSI				
32000 psi, min	43100	43100	42800	40900	39400	42300
	44700	43200	41700	41300	38900	41100
	43900	42500	41700	40900	39000	40300
	43900	43100	41700	40700	40000	40100
	44500	43400	38400	42500	37300	40600
	44300	42800	42100	40400	33800	41700
AVERAGE	44100	43000	41400	41100	38100	41000
		ELONGATION IN 2 IN., PER CENT				
2.5 per cent, min	1.8[a]	1.9[a]	1.8[a]	1.8[a]	1.2[a]	1.8[a]
	2.4[a]	1.8[a]	1.8[a]	1.4[a]	1.5[a]	1.5[a]
	2.0[a]	1.8[a]	1.4[a]	1.2[a]	0.9[a]	1.0[a]
	2.1[a]	1.6[a]	1.9[a]	1.1[a]	1.2[a]	1.0[a]
	2.1[a]	2.0[a]	1.5[a]	1.8[a]	1.4[a]	1.7[a]
	2.3[a]	1.6[a]	1.8[a]	1.8[a]	0.8[a]	1.4[a]
AVERAGE	2.1[a]	1.8[a]	1.7[a]	1.5[a]	1.2[a]	1.4[a]

[a] Low

TABLE VI. - DATA ON ALUMINUM ALLOY NO. ZG61A-T5, SANDCASTING.

ASTM Specification Requirements	3 YR. CONTROL	3- YEARS EXPOSURE				
		STATE COLLEGE	NEW YORK	KURE BEACH	POINT REYES	FREE PORT
		YIELD STRENGTH, 0.2 PER CENT OFFSET, PSI				
25000 psi, min	31000	30200	29700	29400	30100	28600
	31900	29600	29600	29500	28100	30400
	30000	29700	30000	30000	29700	30800
	31200	29700	29500	30100	30500	30500
	31200	29200	29700	29300	29800	30700
	31300	28700	30600	28700	30600	29800
AVERAGE	31100	29500	29900	29500	29800	30100
		TENSILE STRENGTH, PSI				
32000 psi, min	38300	37600	36800	33500	37300	34300
	40400	37200	36700	35500	38500	35900
	37100	37800	38900	36800	34600	37300
	39200	38200	36900	37000	35600	38500
	38700	38100	36900	36400	35200	36600
	38700	36000	37600	34700	36900	35300
AVERAGE	38700	37500	37300	35700	35900	36300
		ELONGATION IN 2 IN., PER CENT				
5.0 per cent, min	3.5	3.5	3.5	2.8[a]	3.7	2.1[a]
	4.9	3.6	3.3	2.7[a]	3.8	2.8[a]
	3.2	4.1	4.8	2.6[a]	2.7[a]	3.6
	4.2	4.1	4.2	3.1	3.0	3.9
	3.6	5.0	4.3	2.8[a]	2.7[a]	2.6[a]
	3.3	3.3	3.6	2.9[a]	2.2[a]	2.6[a]
AVERAGE	3.8	3.9	4.0	2.8[a]	3.0	2.9[a]

[a] Low

TABLE VII.- DATA ON ALUMINUM ALLOY NO. C613A-F, SANDCASTING.-

ASTM Specification Requirements	3 YR. control	3-YEARS EXPOSURE State College	New York	Kure Beach	Point Reyes	Freeport
	YIELD STRENGTH, 0.2 PER CENT OFFSET PSI					
12000 psi, min	23400	23000	22500	23400	22000	23000
	23100	23200	24200	22200	22600	23600
	22600	22100	22800	23700	22800	23700
	21600	22800	22500	23100	23300	23100
	22600	22600	22900	22300	23800	22800
	23400	23000	22600	22500	23000	22600
AVERAGE	22800	22800	22800	23000	22900	23100
	TENSILE STRENGTH, PSI					
19000 psi, min	29800	29000	28200	27900	25000	28700
	28800	28900	29100	25600	26300	27500
	28700	28500	28600	28100	25600	27600
	30300	26600	28800	29800	25200	26900
	29500	29200	26800	27700	27000	27600
	29700	30300	28100	26100	25100	29100
AVERAGE	29500	28800	28300	27300	25700	27900
	ELONGATION IN 2 IN., PER CENT					
1.5 per cent min	1.6	1.8	1.3[a]	1.8	1.2[a]	1.0[a]
	1.6	1.6	1.0[a]	1.2[a]	1.4[a]	0.8[a]
	1.6	1.3[a]	1.5	1.0[a]	1.1[a]	1.6
	1.8	1.7	1.8	1.7	1.4[a]	1.0[a]
	2.4	1.8	1.9	1.3[a]	1.4[a]	0.9[a]
	1.8	1.7	1.2[a]	1.3[a]	0.8[a]	0.9[a]
AVERAGE.	1.8	1.7	1.5	1.4[a]	1.2[a]	1.0[a]

[a] Low

TABLE VIII.- DATA ON ALUMINUM ALLOY NO. 2632A-T5, SANDCASTING.

ASTM Specification Requirements	3 YR. control	3-YEARS EXPOSURE State College	New York	Kure Beach	Point Reyes	Freeport
	YIELD STRENGTH, 0.2 PER CENT OFFSET, PSI					
17000 psi, min	32100	32100	29600	28800	29500	31900
	30800	31000	28400	30500	29400	32000
	31000	30400	30300	30800	30200	32300
	30400	32100	29600	30300	30400	31600
	30600	31500	29600	31300	30700	31100
	31500	30200	30200	29100	29900	31100
AVERAGE	31000	31200	29600	30100	30000	31800
	TENSILE STRENGTH, PSI					
30000 psi, min	40200	39100	36200	37900	36600	36800
	39300	36700	38900	39600	31600	38200
	40800	37000	34800	39300	36500	37800
	41000	34400	39200	36400	37400	38000
	37000	38100	36300	39300	39400	36100
	39600	39000	39500	37800	37000	39000
AVERAGE	39700	37400	38100	38700	36400	37700
	ELONGATION IN 2 IN., PER CENT					
6.0 per cent, min	4.3[a]	4.3[a]	3.2[a]	5.1	3.8[a]	2.6[a]
	4.7[a]	3.1[a]	5.9	5.1	1.5[a]	2.1[a]
	4.3[a]	2.8[a]	4.1[a]	4.3[a]	3.7[a]	3.2[a]
	6.0	2.6[a]	5.8	3.2[a]	3.9[a]	3.5[a]
	3.0[a]	3.8[a]	3.5[a]	4.1[a]	4.1[a]	2.9[a]
	4.0[a]	3.5[a]	4.4[a]	5.4	3.8[a]	3.0[a]
AVERAGE	4.4[a]	3.4[a]	4.5[a]	4.5[a]	3.4[a]	2.8[a]

[a] Low

TABLE IX. - DATA ON ALUMINUM ALLOY NO. 2C81A-T5, SANDCASTING.

TABLE X. - DATA ON ALUMINUM ALLOY No. Z661B-T5, SANDCASTING.

TABLE IX. - DATA ON ALUMINUM ALLOY NO. 2C81A-T5, SANDCASTING.

ASTM Specification Requirements	3 YR. CONTROL	3- YEARS EXPOSURE				
		STATE COLLEGE	NEW YORK	KURE BEACH	POINT REYES	FREE PORT
YIELD STRENGTH 0.2 PER CENT OFFSET PSI						
22000 psi, min	26100	26600	26600	24800	24700	26100
	25400	26400	27900	25000	25800	25400
	26700	26800	26400	24600	26300	27900
	26300	26100	25400	26000	25500	24900
	27400	25600	25000	25800	26400	26100
	26800	—	26000	23800	25100	26000
AVERAGE	26500	26200	26200	25400	25600	26100
TENSILE STRENGTH, PSI						
30000 psi, min	35800	35700	35700	36800	34900	33600
	38000	35900	39000	31800	38000	34300
	37500	39100	36300	33400	36900	36600
	37900	36200	37600	35900	33600	33200
	37700	36200	34600	32000	34300	35900
	36300	—	37200	35800	33000	34300
AVERAGE	37200	36600	36700	34300	35100	34500
ELONGATION IN 2 IN, PER CENT						
3.0 per cent, min	3.8	4.1	4.6	6.0	3.7	2.9[a]
	8.3	4.0	5.1	3.4	5.5	3.4
	5.0	5.7	5.9	3.2	5.4	3.0
	6.2	4.7	5.3	5.4	3.6	3.0
	5.7	5.5	4.8	2.3[a]	3.8	3.6
	4.6	—	5.5	5.3	3.7	3.8
AVERAGE	5.6	4.8	5.2	4.3	4.3	3.3

[a] LOW

TABLE X. - DATA ON ALUMINUM ALLOY No. Z661B-T5, SANDCASTING.

ASTM Specification Requirements	3 YR. CONTROL	3- YEARS EXPOSURE				
		STATE COLLEGE	NEW YORK	KORE BEACH	POINT REYES	FREE-PORT
YIELD STRENGTH, 0.2 PER CENT OFFSET, PSI						
20000 psi, min	34200	33800	34900	33500	34100	34900
	34800	33700	33200	33900	34600	34400
	34700	33300	33900	34200	33500	35000
	35400	33900	33700	34500	34300	35300
	34700	33200	33800	34400	33400	34700
	34300	33600	34400	34000	33700	35200
AVERAGE	34700	33600	34000	34100	33900	34900
TENSILE STRENGTH, PSI						
32000 psi, min	37700	42300	40300	38600	37700	42800
	42300	38600	38000	39600	39800	38800
	40800	40900	39500	40200	39000	40900
	42100	40000	42700	42900	40300	40500
	43200	39000	37700	40500	39800	39500
	38100	39000	39700	41200	40800	39700
AVERAGE	40700	40000	39700	40500	39600	40400
ELONGATION IN 2 IN., PER CENT						
2.0 per cent min	2.5	3.9	2.0	2.6	1.7[a]	3.4
	2.3	2.0	2.2	2.6	2.0	1.9[a]
	2.1	3.8	2.6	2.1	2.4	2.0
	3.3	2.4	4.3	3.5	2.1	1.6[a]
	3.6	2.2	1.8[a]	2.5	2.1	1.9[a]
	1.9[a]	2.7	2.4	3.2	2.8	2.0
AVERAGE	2.7	2.8	2.6	2.8	2.2	2.1

[a] LOW

TABLE XI. - DATA ON ALUMINUM ALLOY NO. 2642 A-T5, SANDCASTING.

ASTM Specification Requirements	3 YR. CONTROL	3-YEARS EXPOSURE STATE COLLEGE	NEW YORK	KURE BEACH	POINT REYES	FREEPORT
		YIELD STRENGTH, 0.2 PER CENT OFFSET, PSI				
22000 psi, min	39300	37800	37000	37300	37400	NO VALUES
	38600	37700	37700	36600	36900	
	38800	37800	37300	37500	36900	
	39800	37200	38000	39400	37700	
	39400	36900	37200	37400	37700	
	39300	37200	37300	37200	36800	
AVERAGE	39200	37300	37400	37600	37200	
		TENSILE STRENGTH, PSI				
33000 psi, min	44000	45300	40700	40300	38700	34100
	41100	43600	41400	39000	39500	38000
	43000	44100	41300	39400	37900	38400
	45500	42200	42200	41000	42300	37000
	44700	41400	41500	39500	41700	36000
	42300	42400	40800	38500	39700	36700
AVERAGE	43400	45200	41300	39700	40000	36700
		ELONGATION IN 2 IN., PER CENT				
2.0 per cent, min	1.8[a]	2.6	1.5[a]	1.3[a]	1.7[a]	0.7[a]
	1.8[a]	2.2	1.7[a]	1.6[a]	1.8[a]	0.7[a]
	1.7[a]	2.1	1.9[a]	1.4[a]	1.4[a]	0.8[a]
	2.6	2.6	1.8[a]	1.5[a]	1.6[a]	1.4
	1.8[a]	1.7[a]	1.6[a]	1.7[a]	2.4	0.8[a]
	1.8[a]	1.8[a]	1.8[a]	1.0[a]	1.7[a]	0.8[a]
AVERAGE	1.9[a]	2.2	1.7[a]	1.4[a]	1.8[a]	0.9[a]

[a] Low

TABLE XII. DATA ON ALUMINUM ALLOY NO. 55B-F, PERMANENT MOLD.

ASTM Specification Requirements	3 YR. CONTROL	3-YEARS EXPOSURE STATE COLLEGE	NEW YORK	KURE BEACH	POINT REYES	FREEPORT
		YIELD STRENGTH, 0.2 PER CENT OFFSET, PSI				
7000 psi, min	11700	12700	12500	10900	14000	12600
	13100	12800	11600	12900	12700	13000
	13100	12200	11800	12000	12100	12100
	12700	11800	11900	10700	13300	11700
	13400	12800	12200	12400	12100	12100
	13400	12100	12600	12600	12200	12200
AVERAGE	12900	10400	12100	11900	12700	12300
		TENSILE STRENGTH, PSI				
21000 psi, min	[illegible]	26500	27800	26000	24800	26400
	27300	27000	27400	26500	24000	26200
	26400	27700	26400	24400	24300	26400
	27600	26600	26500	25800	23500	26400
	27100	27700	27200	26400	24600	27000
	27800	27100	27200	25600	24500	26300
AVERAGE	27400	27100	27100	25800	24300	26500
		ELONGATION IN 2 IN., PER CENT				
2.0 per cent, min	7.1	5.2	7.0	5.1	5.0	5.7
	5.9	5.5	7.0	5.8	4.4	5.0
	5.2	6.0	5.7	4.1	4.9	5.0
	6.9	5.3	5.2	4.6	4.4	5.6
	6.1	7.2	6.9	6.0	4.3	5.3
	6.7	6.8	6.4	4.5	4.7	4.8
AVERAGE	6.3	6.0	6.3	5.0	4.6	5.2

NOTE. - ALL SPECIFICATIONS ON ALUMINUM ALLOY PERMANENT MOLD CASTINGS INCLUDE TABLE II OF ASTM TENTATIVE SPECIFICATIONS B108-56T

TABLE XIII. - DATA ON ALUMINUM ALLOY NO. SC 51A-T6 PERMANENT MOLD.

ASTM

TABLE XIV. - DATA ON ALUMINUM ALLOY NO. SC64B-T61, PERMANENT MOLD.

TABLE XIII. - DATA ON ALUMINUM ALLOY NO. SC 51A-T6 PERMANENT MOLD.

ASTM Specification Requirements	3 YR CONTROL	3-YEARS EXPOSURE				
		STATE COLLEGE	NEW YORK	KURE BEACH	POINT REYES	FREE-PORT
YIELD STRENGTH, 0.2 PER CENT OFFSET, PSI						
23 000 psi, min	29100	28600	31300	31900	29000	32900
	32800	28200	30400	31000	32100	32000
	31400	28000	29300	28500	30600	29900
	29900	27100	27400	32100	28200	32200
	31400	28600	28200	29400	27800	31200
	30700	—	31000	30600	30800	31500
AVERAGE	30900	28100	29600	30500	29800	31600
TENSILE STRENGTH, PSI						
37000 psi, min	41400	44600	43900	43700	40900	43700
	38700[b]	41800	45300	43300	41800	44200
	44200	44300	44600	44100	41200	42200
	38600[b]	46300	43100	43000	40200	41800
	39100[b]	43300	44700	43400	39300	43000
	45400	—	44400	42300	40200	44800
AVERAGE	41200	44100	44300	43300	40600	43300
ELONGATION IN 2 IN., PER CENT						
1.5, PER CENT min.	2.9	5.0	4.2	3.8	3.3	2.8
	1.6[b]	2.4	5.4	3.8	2.8	3.5
	3.2	4.4	4.3	4.8	2.9	3.5
	2.4[b]	5.6	4.5	3.5	3.3	2.1
	1.9[b]	4.0	4.9	3.8	2.6	3.1
	4.9	—	4.8	3.4	3.4	4.2
AVERAGE	2.8	4.3	4.7	3.9	3.1	3.2

b - GAS INCLUSIONS NOTED IN SAMPLES

TABLE XIV. - DATA ON ALUMINUM ALLOY NO. SC64B-T61, PERMANENT MOLD.

ASTM Specification Requirements	3 YR. CONTROL	3-YEARS EXPOSURE				
		STATE COLLEGE	NEW YORK	KURE BEACH	POINT REYES	FREE-PORT
YIELD STRENGTH, 0.2 PER CENT OFFSET, PSI						
26000 psi, min	36300	35100	35100	34400	34600	36600
	37200	35500	37900	35900	34700	36100
	37600	35200	35200	38100	36100	37300
	36800	35500	38400	34600	34200	36500
	36000	35200	37500	35300	36400	37700
	39000	35700	36400	34700	35700	37600
IVERAGE	37200	35300	36800	35500	35300	37000
TENSILE STRENGTH, PSI						
40000 psi, min	44700	44200	44200	42200	37700[a]	42200
	44200	44200	45700	44900	39000[a]	42700
	44900	43900	43700	41300	38100[a]	44500
	44100	45100	44800	42800	38400[a]	43500
	44900	44300	45400	43200	39600[a]	43300
	45600	44300	44500	42800	36100	41800
AVERAGE	44700	44300	44700	42900	38200	43000
ELONGATION IN 2 IN., PER CENT						
2.0 per cent, min	1.7[a]	1.3[a]	2.9	1.2[a]	0.6[a]	0.9[a]
	1.6[a]	1.5[a]	1.6[a]	1.6[a]	0.9[a]	1.0[a]
	1.9[a]	1.7[a]	1.5[a]	1.4[a]	0.6[a]	1.0[a]
	1.4[a]	1.6[a]	1.7	1.1[a]	0.9[a]	1.2[a]
	1.8[a]	1.6[a]	1.8[a]	1.7[a]	1.0[a]	0.8[a]
	1.5[a]	1.7[a]	2.2	1.6[a]	0.7[a]	0.7[a]
AVERAGE	1.7[a]	1.6[a]	1.9[a]	1.5[a]	0.8[a]	0.9[a]

[a] LOW

TABLE XV. - DATA ON ALUMINUM ALLOY NO. ZG 32A-T5, PERMANENT MOLD.

ASTM Specification Requirements	3 YR. CONTROL	3- YEARS EXPOSURE				
		STATE COLLEGE	NEW YORK	KURE BEACH	POINT REYES	FREE-PORT
YIELD STRENGTH, 0.2 PER CENT OFFSET, PSI						
17000 psi, min	34200	33700	33400	34000	33500	35100
	35100	32400	33500	32900	33300	34800
	34900	32800	32900	33200	32700	-
	35200	33300	33000	34000	32900	35300
	34300	32500	32400	33000	33300	34000
	34700	33100	—	33600	33100	33900
AVERAGE	34700	33000	33000	33500	33100	34600
TENSILE STRENGTH, PSI						
37000 PSI, min	52200	51700	51600	52000	46600	48700
	53200	50300	51500	50700	51400	49700
	52800	48600	51800	45000	48300	—
	52600	52500	50500	52200	50200	50900
	52400	50600	51000	49300	49100	48100
	51800	50600	—	50800	50400	47400
AVERAGE	52500	50700	51300	50000	49300	49000
ELONGATION IN 2 IN., PER CENT						
10.0 PERCENT, min	12.2	12.4	12.1	11.7	6.2[a]	6.3[a]
	11.3	11.0	12.1	10.4	10.7	7.6[a]
	11.7	10.5	12.7	5.5	8.4[a]	—
	11.3	12.6	11.3	11.0	8.7[a]	7.4[a]
	11.6	11.6	11.8	9.0[a]	8.6[a]	5.7[a]
	10.7	11.0	—	10.3	10.8	5.8[a]
AVERAGE	11.5	11.5	12.0	9.7[a]	8.9[a]	6.6[a]

[a] LOW

TABLE XVI. - DATA ON ALUMINUM ALLOY NO. ZC60A-T5, PERMANENT MOLD.

ASTM Specification Requirements	3 YR. CONTROL	3-YEARS EXPOSURE				
		STATE COLLEGE	NEW YORK	KURE BEACH	POINT REYES	FREE-PORT
YIELD STRENGTH, 0.2 PER CENT OFFSET, PSI						
18000 psi, min	23500	23100	23300	23700	23400	22800
	22900	23600	22900	23900	21500	22800
	23900	23200	27300	22400	23600	23800
	24500	23000	23300	23700	23300	24500
	23800	23300	23100	23900	22600	22800
	22300	—	—	24400	23600	24500
AVERAGE	23500	23200	24000	23700	23000	23500
TENSILE STRENGTH, PSI						
28000 psi, min	40900	35600	39000	37700	39300	38400
	38100	38400	38500	39800	33100	37500
	40600	37800	38300	33900	39000	40600
	42400	36000	37800	38000	37300	39700
	40600	40000	39000	39100	35600	39100
	40500	—	—	39600	36400	39200
AVERAGE	40500	37600	38500	38000	36800	39200
ELONGATION IN 2 IN., PER CENT						
7.0 PERCENT min	9.7	5.7[a]	8.2	6.7[a]	9.1	7.0
	6.6	7.8	8.4	7.9	5.2[a]	6.4[a]
	9.7	7.6	1.3	5.6[a]	7.4	10.6
	10.9	9.1	7.5	7.6	7.2	7.4
	9.8	9.2	9.1	8.1	6.0[a]	7.9
	10.4	—	—	8.4	6.3[a]	7.8
AVERAGE	9.5	7.9	8.1	7.4	7.2	7.7

[a] Low

TABLE XVII. - DATA ON ALUMINUM ALLOY NO. ZC 81B-T5, PERMANENT MOLD.

TABLE XVIII. - DATA ON ALUMINUM ALLOY No. ZG 42A-T5, PERMANENT MOLD.

TABLE XVII. - DATA ON ALUMINUM ALLOY NO. ZC 81B-T5, PERMANENT MOLD.

ASTM Specification Requirements	3 YR. Control	3-YEARS EXPOSURE State College	New York	Kure Beach	Point Reyes	Free Port
YIELD STRENGTH, 0.2 PER CENT OFFSET, PSI						
22000 psi, min	25600	24500	23800	24500	28500	26200
	25100	26500	25100	25100	25700	26100
	25400	26500	27400	24400	25700	22400
	21000	27700	25500	22000	24500	26300
	—	27700	24500	27000	25000	26500
	—	26000	—	24200	24800	—
AVERAGE	24300	26800	25300	24500	25700	25500
TENSILE STRENGTH, PSI						
32000 psi, min	36900	30200[a]	32300	30300[a]	36800	33300
	37500	31700[a]	36000	35800	36500	37700
	34000	39100	39100	33600	35700	32300
	37300	38800	35900	31500[a]	32100	37500
	—	39400	35500	36500	31100[a]	37000
	—	40000	—	38100	32800	—
AVERAGE	36400	36500	35800	34300	34200	35600
ELONGATION IN 2 IN., PER CENT						
4.0 per cent, min	4.4	2.0[a]	3.5[a]	1.9[a]	2.6[a]	2.5[a]
	5.7	1.2[a]	4.3	4.1	3.8[a]	4.4
	3.6[a]	5.9	5.0	3.5[a]	3.5[a]	—
	4.0	4.0	3.9[a]	2.0[a]	2.5[a]	4.3
	—	5.2	3.4[a]	4.1	2.2[a]	3.6[a]
	—	5.1	—	4.0	2.9[a]	—
AVERAGE	4.4	3.9[a]	4.0	3.3[a]	2.9[a]	3.7[a]

[a] Low

TABLE XVIII. - DATA ON ALUMINUM ALLOY No. 26 42A-T5, PERMANENT MOLD.

ASTM Specification Requirements	3 YR. Control	3-YEARS EXPOSURE State College	New York	Kure Beach	Point Reyes	Free-Port
YIELD STRENGTH, 0.2 PER CENT OFFSET PSI						
25000 psi, min	44100	42000	42700	42000	42500	43800
	43200	42300	42700	42400	44600	44800
	44800	41500	44000	42100	41900	43400
	44000	42000	43000	42100	41800	44300
	45700	41700	42000	42400	42200	45600
	44300	42000	41800	42600	43700	44900
AVERAGE	44400	41900	42700	42300	42800	44500
TENSILE STRENGTH, PSI						
42000 psi, min	51100	50100	52000	49600	54400	46500
	52700	49800	51200	50200	50400	49000
	53200	50000	48900	48200	49700	47200
	48000	50300	52300	48900	48800	48600
	52000	51600	51300	51300	48400	52000
	52100	49400	50500	49100	51100	46300
AVERAGE	51500	50200	50900	49600	50500	48300
ELONGATION IN 2 IN., PER CENT						
4.0 per cent, min	2.0[a]	2.4[a]	3.1[a]	2.6[a]	2.2[a]	0.8[a]
	2.8[a]	2.3[a]	3.2[a]	1.8[a]	1.8[a]	0.8[a]
	2.7[a]	2.6[a]	1.7[a]	1.8[a]	2.6[a]	0.7[a]
	2.2[a]	2.3[a]	3.5[a]	1.8[a]	1.7[a]	1.1[a]
	1.4[a]	3.2[a]	3.4[a]	2.7[a]	1.8[a]	1.8[a]
	2.0[a]	2.2[a]	3.1[a]	2.4[a]	2.2[a]	0.8[a]
AVERAGE	2.2[a]	2.5[a]	3.0[a]	2.2[a]	2.1[a]	1.0[a]

[a] Low

TABLE XIX. - DATA ON MAGNESIUM ALLOY NO. AZ63A-T4, SANDCASTING.

ASTM Specification Requirements	3YR. CONTROL	3-YEARS EXPOSURE STATE COLLEGE	NEW YORK	KURE BEACH	POINT REYES	FREE PORT
	YIELD STRENGTH 0.2 PER CENT OFFSET, PSI					
NOT REQUIRED	17300	15700	14800	17100	15700	17500
	17000	16200	16500	16700	16000	17500
	16800	15700	16600	18100	15700	18500
	17400	16300	16500	17300	16300	16900
	18200	16900	15300	17400	16900	17500
	17800	16700	16200	17000	16600	17600
AVERAGE	17400	16300	16000	17300	16300	17600
	TENSILE STRENGTH, PSI					
34000 PSI, min	42300	39200	35800	40000	39500	40100
	41000	40800	38100	41400	39100	40500
	39700	38800	38400	40200	39300	41300
	40600	40300	39400	39700	40400	40900
	40900	39900	38600	40300	41100	40500
	42600	37200	39000	39300	38200	39800
AVERAGE	41200	39400	38200	40200	39600	40500
	ELONGATION IN 2IN., PER CENT					
7.0 percent, min	15.6	13.2	10.0	13.1	14.4	10.8
	13.2	15.5	13.1	15.8	12.9	12.0
	11.6	11.9	13.5	11.4	13.2	11.9
	13.5	14.5	13.4	12.8	14.7	13.3
	12.3	12.6	12.8	13.6	16.6	11.3
	16.7	8.9	13.8	13.1	11.9	11.4
AVERAGE	13.8	12.8	12.8	13.3	14.0	11.8

NOTE - ALL SPECIFICATIONS ON MAGNESIUM ALLOY SANDCASTINGS INCLUDE TABLE II OF ASTM TENTATIVE SPECIFICATIONS B 80 - 56T

TABLE XX.- DATA ON MAGNESIUM ALLOY NO. AZ91C-F, SANDCASTING.

ASTM Specification Requirements	3YR CONTROL	3-YEARS EXPOSURE STATE COLLEGE	NEW YORK	KURE BEACH	POINT REYES	FREE PORT
	YIELD STRENGTH, 0.2 PER CENT OFFSET, PSI					
NOT REQUIRED	17200	17200	16700	17500	17700	17600
	19600	18300	15200	18600	17500	17100
	18800	17200	15800	18000	18200	18700
	18300	17800	17000	18200	19300	17400
	19000	17900	15500	18600	18600	16800
	18600	18100	17000	18400	18000	17800
AVERAGE	18600	17800	16200	18200	18200	17600
	TENSILE STRENGTH, PSI					
20000 psi, min	25800	24000	24000	24400	22900	24000
	26800	25800	21900	24500	22800	23400
	25700	24400	22800	22800	23500	26100
	24900	24800	24400	23300	24900	23800
	26300	24500	21400	23900	24100	23900
	26100	25800	23900	23900	22500	24900
AVERAGE	25900	24900	23100	23800	23500	24400
	ELONGATION IN 2IN., PER CENT					
NOT REQUIRED	2.2	2.6	3.2	2.2	2.1	1.7
	2.2	2.2	1.8	1.8	1.5	1.7
	2.5	2.2	2.8	1.3	1.3	1.7
	3.1	2.3	3.0	1.6	1.8	1.6
	3.2	2.1	2.2	1.8	2.0	1.9
	3.1	2.4	2.4	1.4	1.0	2.0
AVERAGE	2.7	2.3	2.6	1.7	1.6	1.8

TABLE XXI. - DATA ON MAGNESIUM ALLOY NO. AZ 92A-T6, SANDCASTING.

TABLE XXII. - DATA ON MAGNESIUM ALLOY NO. EK 30A-F, SANDCASTING.

TABLE XXI. - DATA ON MAGNESIUM ALLOY NO. AZ 92A-T6, SANDCASTING.

ASTM Specification Requirements	3 YR CONTROL	3 – YEARS EXPOSURE STATE COLLEGE	NEW YORK	KURE BEACH	POINT REYES	FREE PORT
YIELD STRENGTH, 0.2 PER CENT OFFSET, PSI						
18000 psi, min	23000	23600	22000	23400	22700	20500
	24000	23200	20600	24000	21200	20100
	22700	23500	20700	23300	22500	21600
	22200	21700	21100	23200	22100	20100
	23400	21900	23700	23700	23100	20900
	21800	22900	21300	21600	22400	21400
AVERAGE	22900	22800	21600	23200	22300	20800
TENSILE STRENGTH, PSI						
34000 psi, min	41600	42600	40500	41000	40500	37300
	38600	39400	34100	39000	39100	39700
	42400	39200	39800	40300	39600	37500
	36600	41300	39700	40700	39200	36000
	42100	39000	39700	38100	39500	38200
	38500	41900	39700	39000	38300	36400
AVERAGE	40000	40600	38600	39700	39400	37500
ELONGATION IN 2 IN., PER CENT						
1.0 per cent, min	4.1	4.9	4.5	4.2	4.8	2.2
	3.0	2.7	3.3	3.3	3.0	2.6
	4.3	2.9	5.2	3.8	3.6	2.6
	2.4	4.7	3.9	3.8	3.2	2.4
	4.3	2.8	3.8	3.2	3.3	2.8
	2.9	5.0	3.3	3.8	3.3	2.1
AVERAGE	3.5	3.8	4.0	3.7	3.5	2.5

TABLE XXII. - DATA ON MAGNESIUM ALLOY NO. EK 30A-F, SANDCASTING.

ASTM Specification Requirements	3 YR. CONTROL	3 YEARS EXPOSURE STATE COLLEGE	NEW YORK	KURE BEACH	POINT REYES	FREE-PORT
YIELD STRENGTH, 0.2 PER CENT OFFSET, PSI						
NOT REQUIRED	12800	13300	11700	13000	12600	12300
	13100	11800	12100	12700	11200	12800
	12800	12400	11600	12600	13000	12700
	12400	14000	11800	12600	12900	12300
	12400	14100	12000	13000	11800	12500
	13600	13800	–	12500	12500	11300
AVERAGE	12900	13200	11800	12700	12300	12300
TENSILE STRENGTH, PSI						
NOT REQUIRED	17800	17700	15400	17600	17200	17000
	19100	17900	17700	17600	15000	17000
	18400	17600	16900	17700	17800	17500
	17800	19400	16100	18000	17400	16000
	18400	19200	17200	17900	15700	16700
	18900	19700	–	17000	17000	16700
AVERAGE	18400	18600	16700	17600	16700	16800
ELONGATION IN 2 IN., PER CENT						
NOT REQUIRED	2.5	1.6	1.8	1.8	3.0	1.3
	2.9	2.3	2.7	1.7	2.5	1.4
	2.2	1.4	2.6	1.8	2.6	1.9
	2.2	2.3	2.0	2.0	3.2	1.3
	2.8	2.3	2.9	2.2	1.8	1.5
	2.1	2.1	–	1.9	2.3	1.4
AVERAGE	2.5	2.0	2.4	1.9	2.6	1.5

TABLE XXIII. - DATA ON MAGNESIUM ALLOY NO. EK30A-T6, SANDCASTING.

ASTM Specification Requirements	3 YR. CONTROL	3-YEARS EXPOSURE				
		STATE COLLEGE	NEW YORK	KURE BEACH	POINT REYES	FREE-PORT
YIELD STRENGTH, 0.2 PER CENT OFFSET, PSI						
14000 psi, min	15100	16700	16400	16700	15000	14500
	15500	16500	15400	16300	14200[a]	14400
	16300	17000	14900	17100	13800	14100
	17000	16100	15900	16100	14200	15100
	14600	16600	15300	15900	14600	15300
	—	15900	15900	17200	14400	14900
AVERAGE	15700	16500	15600	16600	14400	14700
TENSILE STRENGTH, PSI						
20000 psi, min	22200	21800	24100	21900	20400[a]	18700[a]
	22600	22300	20800	21300	19700[a]	19600[a]
	24100	23100	21600	23600	19000[a]	20100
	20500	23100	22500	21500	19000[a]	19800[a]
	21500	23200	20700	23400	19000[a]	20300
	—	22200	23000	22900	18100[a]	20900
AVERAGE	22300	22600	22100	22400	19200	19900
ELONGATION IN 2 IN., PER CENT						
2.0 per cent, min	3.0	1.7[a]	4.8	1.9[a]	2.2	1.0[a]
	3.0	2.3	2.0	2.6	2.0	0.8[a]
	2.9	2.8	3.2	3.4	1.4[a]	1.5[a]
	3.5	3.1	3.6	2.4	1.8[a]	1.3[a]
	1.7[a]	3.1	1.5[a]	3.8	1.4[a]	1.5[a]
	—	2.6	3.6	2.6	1.4[a]	2.1
AVERAGE	2.8	2.6	3.1	2.8	1.7[a]	1.4[a]

[a] Low

TABLE XXIV. - DATA ON MAGNESIUM ALLOY NO. AZ91C-T6, SANDCASTING.

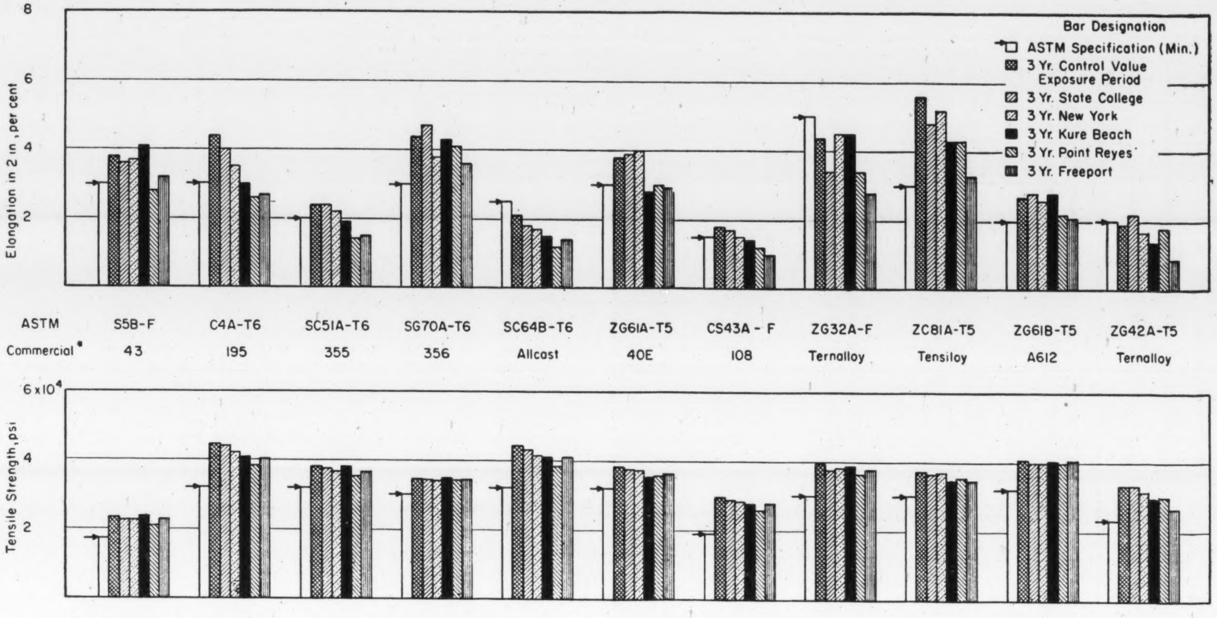
ASTM Specification Requirements	3 YR. CONTROL	3-YEARS EXPOSURE				
		STATE COLLEGE	NEW YORK	KURE BEACH	POINT REYES	FREE-PORT
YIELD STRENGTH, 0.2 PER CENT OFFSET, PSI						
16000 psi, min	20200	21300	21200	20400	21000	19900
	19800	20400	20300	22500	20000	17700
	20700	22500	19400	20400	19800	20200
	19700	17900	19900	22600	19500	19800
	19800	17800	19800	22400	22000	21000
	21600	19400	19900	20100	20300	—
AVERAGE	20300	19900	20100	21400	20700	19700
TENSILE STRENGTH, PSI						
34000 psi, min	41200	37300	39600	36500	35400	37100
	35300	37200	37100	37900	38100	36200
	41500	39300	36000	32800[a]	35700	38100
	41100	34700	37500	36100	36600	37200
	42800	35000	38700	36400	38000	34700
	41600	36600	38500	35200	35700	—
AVERAGE	40600	36700	37900	35800	36600	36700
ELONGATION IN 2 IN., PER CENT						
3.0 per cent, min	5.5	3.4	5.0	4.9	3.1	3.8
	2.9[a]	3.6	3.4	3.5	3.4	3.1
	4.5	4.2	3.4	3.4	3.0	5.7
	4.8	5.5	4.2	4.7	4.4	4.3
	5.4	4.7	4.5	3.5	3.4	2.3[a]
	4.1	4.7	3.8	4.2	3.0	—
AVERAGE	4.5	4.4	4.1	4.0	3.3	3.8

[a] Low

TABLE XXV. — DATA ON MAGNESIUM ALLOY NO. EZ33A-T5, SANDCASTING.

ASTM Specification Requirements	3YR. CONTROL	3-YEARS EXPOSURE				
		STATE COLLEGE	NEW YORK	KURE BEACH	POINT REYES	FREE-PORT
		YIELD STRENGTH, 0.2 PER CENT OFFSET, PSI				
14000 psi, min	15200	13500[a]	14700	13800[a]	13500[a]	14800
	15200	13700[a]	13900[a]	13500[a]	14400	15000
	15400	13100[a]	14400	13800[a]	13800[a]	14900
	15100	13200[a]	13800[a]	13600[a]	13000[a]	16000
	15200	13900[a]	15300	13200[a]	13500[a]	15000
	15100	13700[a]	13800[a]	13900[a]	13700[a]	15300
AVERAGE	15200	13500[a]	14300	13600[a]	13700[a]	15200
		TENSILE STRENGTH, PSI				
20000 psi, min	20700	15700[a]	20100	18700[a]	17100[a]	20300
	21300	15400[a]	18600[a]	17700[a]	18000[a]	20200
	20500	18900[a]	19000[a]	17500[a]	19900[a]	23100
	20400	18300[a]	18600[a]	17200[a]	17600[a]	19100[a]
	19800[a]	18300[a]	20100	18300[a]	15800[a]	21400
	20600	16100[a]	20100	17800[a]	18000[a]	19800[a]
AVERAGE	20600	17100[a]	19400[a]	17900[a]	17700[a]	20700
		ELONGATION IN 2IN., PER CENT				
2.0 per cent, min	2.1	0.8[a]	2.2	1.8[a]	1.3[a]	2.1
	2.2.	0.6[a]	2.6	1.3[a]	2.1	1.8[a]
	2.3	1.6[a]	1.6[a]	1.3[a]	1.8[a]	2.1
	1.5[a]	2.3	2.3	1.5[a]	1.3[a]	0.8[a]
	2.2	2.1	2.3	1.8[a]	1.0[a]	1.9[a]
	2.2	1.2[a]	2.8	1.5[a]	1.5[a]	1.3[a]
AVERAGE	2.1	1.4[a]	2.3	1.5[a]	1.5[a]	1.7[a]

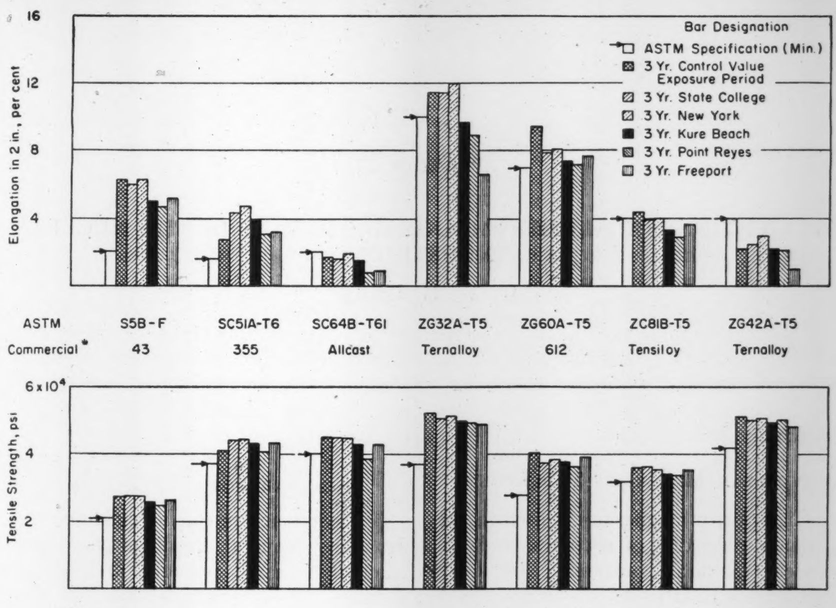
[a] LOW



*As Most Commonly Used.

Each Bar Represents Average Results on Six Specimens.

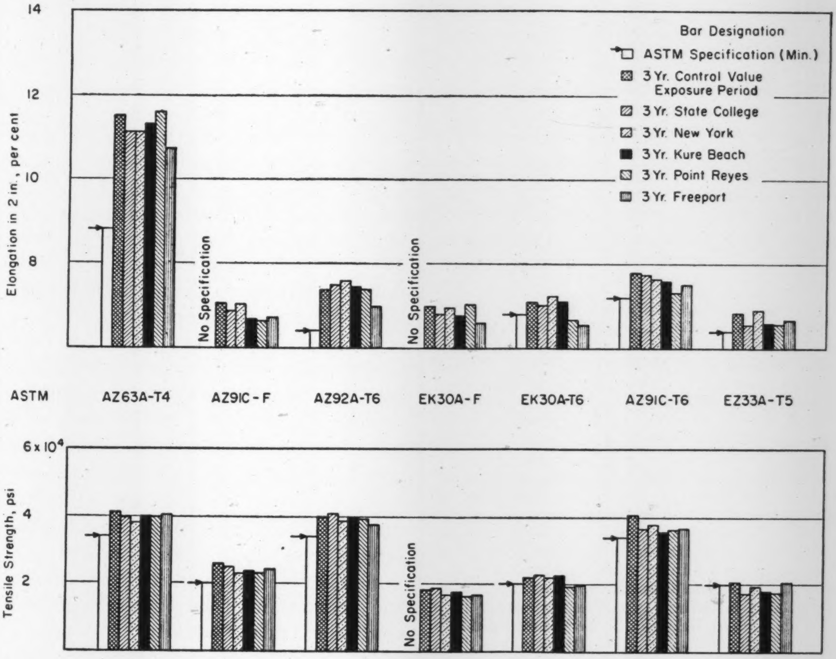
FIG. 1.—Summary of Data, Atmospheric Exposure of Light Metals and Alloys, Aluminum Alloy Sandcastings.



*As Most Commonly Used.

Each Bar Represents Average Results on Six Specimens.

FIG. 2.—Summary of Data, Atmospheric Exposure of Light Metals and Alloys, Aluminum Alloy Permanent Mold.



Each Bar Represents Average Results on Six Specimens.

FIG. 3.—Summary of Data, Atmospheric Exposure of Light Metals and Alloys, Magnesium Alloy Sand Castings.

APPENDIX III

APPLICATIONS OF ALUMINUM AND MAGNESIUM IN THE NUCLEAR INDUSTRIES*

By W. G. Groth[1]

The segments of the nuclear energy field of interest to ASTM Committee B-7 are nuclear materials processing, nuclear power reactors, and nuclear research and special reactors.[2]

Nuclear Materials Processing

High-purity magnesium is used in the primary reduction of uranium, and the nuclear materials processing industries use large quantities of aluminum alloys in both tubing and plate form for the transfer and containment of chemicals and materials in process. In much of this work the actual corrosion rate of pipe, for example, when considered from a replacement standpoint, is less important than the possibility of contaminating process fluids with corrosion products. It is also important that the alloys used for structural purposes be amenable to fabrication by fusion welding in an inert gas atmosphere. The principal alloys include 1100, 3003, 5052, and 6061 aluminum, and it is not likely that this picture will change appreciably in the foreseeable future. Industry has found the standard commercial alloys produced to current specifications entirely satisfactory for most of this service.

Nuclear Power Reactors

Nuclear reactors can be classified under three general headings as power reactors, research reactors, and special reactors. Power reactors are those nuclear energy sources utilized as all or part of the heat or energy input section of central power stations producing electrical or other energy in the megawatt range. The term "power generation" implies the generation of vast quantities of heat with the simultaneous conditions of high temperatures and high pressures. These conditions in turn encourage visualization of massive installations of great physical weight. Up to the present time, aluminum and magnesium have had a very small place in the field of power generation except for conductors; and aluminum has not usually been used for bus installations except in times of national emergency. The primary temperatures and pressures required for high thermal efficiency of our larger industrial steam cycle power plants have been beyond the capabilities of aluminum and magnesium alloys. There are, however, certain peculiarities of nuclear materials and reactions which have largely prevented utilization of these same high temperatures and pressures in the generation of heat power through controlled nuclear activity.

In the consideration of light metals

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* Presented at the meeting of Committee B-7 held in St. Louis, Mo., Feb. 1958.

[1] Senior Welding Engineer, ACF Industries, Inc., Albuquerque, N. Mex.

[2] In most fields, aluminum is used because of its light weight, resistance to corrosion in certain media, workability and good appearance. Magnesium has been accepted and eventually required in many industries for one or more of the same reasons, although not necessarily the same specific reason in many instances. In the nuclear fields it would not be expected that weight would be a factor except in the instance of air or waterborne power plants (including transportation of components of military power reactors) or certain articles of military ordnance. Even in these instances it is entirely possible that other light metals such as titanium or beryllium would be more desirable on a strength-to-weight basis where cost is not a factor.

for nuclear reactor applications, one might well establish two fields of investigation, namely (1) internal reactor components, and (2) external components. The internal or integral components include fuel elements and reactor shells, in which the prime considerations are (*a*) service temperature, (*b*) reaction between material and the nuclear fuel, (*c*) reaction between the material and the reactor coolant, (*d*) thermal neutron cross-section, (*e*) thermal conductivity, and (*f*) welding characteristics. Table I lists some of the more important properties, including neutron absorption, of a number of reactor materials.

TABLE I.—PROPERTIES OF REACTOR MATERIALS.

	Aluminum	Beryllium	Magnesium	Molybdenum	Titanium	Zirconium	18 8 Stainless Steel (Fe, Cr, Ni, Mn, Cb)
Density, g per cu cm	2.70	1.85	1.74	10.2	4.5	6.5	7.92
Melting point,							
deg Cent	660	1300	651	2620	1725	1830	1399 to 1421
deg Fahr	1220	2370	1205	4750	3135	3325	2570
Thermal expansion coefficient per deg Cent × 10^6	24	12	26	5.5	8.5	4.96 to 5.8	16.7
Thermal conductivity, cal per sq cm per sec per deg Cent per cm	0.54	0.3	0.35	0.346	low	0.05	0.039
Modulus of elasticity, 10^6 psi	10	42	6.5	40 to 50	16.8	12	29
Tensile strength, 10^3 psi							
Annealed	13	45	32 to 46	100	80	35	90
Cold worked	24		32 to 50	250	122	85	...
Temperature above which creep is an important factor, deg Cent	150	700	170	850	...	500	700
Thermal neutron absorption cross-section, 10^3 σ abs cm^2 per cm^3	13	1.23	2.54	...	...	7.65	200+

Thermal Neutron Cross-Section:

One of the principal advantages of aluminum and magnesium (and incidentally beryllium and zirconium) for use incide nuclear reactors is their low thermal neutron absorption cross-section when compared with stainless steel and other common high density materials. It is most desirable that this value be as low as possible so that the canning materials or other internal parts not specifically designated as shielding or control elements have a minimum effect upon the nuclear activity used for heat generation. In other words, neutrons absorbed by fuel element cans, for instance, are not available to help sustain nuclear fission; therefore this canning material must be compensated for by increasing the amount of nuclear fuel either through larger charges or higher enrichment where enriched fuel is used, increasing reactor size, fuel cost, and control problems.

Much developmental work has been done on both the aluminum and magnesium alloy systems to find better materials for internal reactor components. Many aluminum alloys have been tested for this type of service, since aluminum has excellent formability, is of low cost, and possesses good nuclear properties.

Mechanical Properties:

While the thermal neutron absorption cross-section of aluminum is many times that of magnesium, it does have better thermal conductivity and a higher modulus of elasticity. Most of the alloy developmental work on aluminum appears to have centered around aluminum nickel alloys (for example, AA alloy X8001, a 1 per cent nickel alloy) and ultra-high-purity aluminum (AA alloy 1245). The aluminum-nickel alloys are being tested for use in water-cooled reactors and have apparently become of sufficient importance to warrant attention by the various metals specification committees. The term "high-purity," as used today for alloys containing more than 99.7 per cent aluminum, may be a misnomer since certain British investigators have evaluated the effects of micro quantities of certain residuals and have, in one instance, referred to a "0.1 per cent silicon alloy." It may be that elements considered residuals a few years ago will be revealed as worthy of precise control in nuclear applications. The above statement is true for both aluminum and magnesium when weldability is a consideration, as is always the case in the fabrication of fuel elements. Micro quantities of silicon and iron have been found to have considerable effect upon the cracking tendency of high-purity aluminum.

Several magnesium alloys have been developed to improve the strength and creep properties at reactor service temperatures. The English report an alloy (Magnox A-12, a magnesium, 0.75 per cent aluminum plus beryllium alloy) which shows many desirable characteristics under test conditions, including wet carbon dioxide at 615 C (1130 F). Magnesium has been alloyed with a number of other metals, including calcium, aluminum, and manganese. Many experimental alloys have proved unsatisfactory because of oxidation resistance, stress corrosion, alloy decomposition, or lack of weldability. High-purity magnesium (available in purities of 99.95 and possibly 99.97 per cent) has a number of advantages over magnesium alloys, but in view of its low oxidation resistance and low strength at elevated temperature, certain of the alloys are more desirable for many applications. Where magnesium alloys are used as a canning material it is common practice to provide a mechanical bond between the can and fuel element. This is done to prevent destruction of the can from repeated cycling and "ratcheting" resulting from differential thermal expansion between the two materials. The desirability of magnesium in this application is best exemplified by Great Britain's Calder Hall Reactor, and modifications of this system are now proposed for many nuclear power plants throughout the world (including our GCPR project).

TABLE II.—REACTION STARTING TEMPERATURE OF URANIUM AND CANNING MATERIAL.

Aluminum	forms compound at 175 C (348 F)	melts at 660 C (1220 F)
Magnesium	no reaction	melts at 650 C (1202 F)
Beryllium	approximately 600 C (1112 F)	melts at 1300 C (2370 F)
Steel	melts at 725 C (1337 F)	alloys above 550 C (1022 F)
Zirconium	slow action at 800 C (1472 F)	rapid above melting point
Tantalum	attacks at melting point	rapid above 1300 C (2372 F)
Tungsten	rapid above 1400 C (2550 F)	

In the welding of magnesium it has been determined that the calcium content can be most troublesome, and the specifications for magnesium alloys for reactor service may restrict calcium content to a much lower value than is customary in current industrial specifications. ASTM specifications already reflect this thinking in changing calcium limits from approximately 0.6 per cent to 0.04 per cent maximum or less when welding is required.

TABLE III.—ONSET OF REACTION BETWEEN COOLANT AND CAN.

	Aluminum	Magnesium	Stainless Steel	Zirconium	Beryllium	Tantalum
Air	safe to melting point; 640 C (1180 F)	300 C (570 F)	500 C (932 F)	300 C (570 F)	800 C (1472 F)	300 C (570 F)
Water	150 C pure; 300 C alloyed Fe and Ni	70 C (158 F)	500 C (932 F)	320 C (608 F) Alloy with Sn, Ni	unsuitable	300 C (570 F)
Carbon dioxide	melting point	550 C (1020 F)	500 C (932 F)	450 C (840 F)	550 C (higher if dry)	....
Hydrogen	safe to melting point; permeable at high temperature	450 C (840 F); permeable at high temperature	may embrittle under irradiation	150 C (302 F)	600 C (1112 F)	....
Helium	no action	no action	no action	no action	no action	no action
Sodium	attacks	attacks	800 C (1472 F)	oxide free—no action; oxide present, 250 C (482 F)	....	oxide free—no action; oxide present, 250 C
Bismuth	attacks	attacks	400 C, 550 C with zirconium (752 F)	attacks, 600 C (1112 F)	attacks	600 C (1112 F)
Mercury	attacks	attacks	400 C (752 F)	attacks	attacks	500 C (932 F)

Reaction Between Material and Nuclear Fuel:

Table II outlines the metallurgical reactions that can be expected between conventional uranium fuel alloys and a variety of canning materials. It is immediately evident that aluminum has disadvantages because of the formation of a compound of aluminum and uranium at 175 C (348 F), but this can be inhibited with some success by using buffer compounds between the aluminum and the uranium fuel material. These buffers include an aluminum-silicon alloy and pure nickel. Unfortunately this silicon alloy has low ductility and a low melting point, while nickel is a neutron absorber. Magnesium does not react with the fuel material, but the service temperature is limited by the melting temperature (as is the case with aluminum). In both instances the melting temperature is somewhat below that of the other materials listed. In any event, the temperature of the fuel element is limited to about 550 C (1020 F) in both instances. Table III shows the maximum allowable temperatures between various canning materials and a number of reactor coolants.[3]

Reaction Between Material and Reactor Coolant:

It will be noted that aluminum and magnesium are satisfactory canning materials only when used with nonmetallic reactor coolants. Aluminum is also serviceable in the case of water-cooled reactors at temperatures below 300 C (570 F), making it an acceptable canning material for research, training, and other special low-power water-cooled reactors. In the case of gas-cooled reactors, it would appear that either aluminum or magnesium alloys are serviceable to the maximum temperatures attainable with many other canning materials. As noted before, certain of the gas-cooled reactors fabricated in England utilize a magnesium alloy as the fuel element can with an apparently satisfactory service record.

TABLE IV.—INDUCED ACTIVITIES IN METALS PRESENT IN STRUCTURAL MATERIALS, GAMMA RADIOACTIVITY RESULTING FROM NEUTRON BOMBARDMENT.

(Compiled from various sources)

Element	Isotope Mass Number	Natural Abundance, per cent	Active Species	Half-Life	X-ray Energy, Mev
Titanium	50	5.3	Ti^{51}	6 minutes	0.32
Chromium	50	4.4	Cr^{51}	27 days	0.32
Manganese	55	100.0	Mn^{56}	2.6 hours	2.1
Iron	58	0.33	Fe^{59}	45 days	1.3
Cobalt	59	100.0	Co^{60}	5.3 years	1.3
Nickel	64	1.9	Ni^{65}	2.6 hours	1.5
Copper	63	69.0	Cu^{64}	12.8 hours	1.34
Zinc	64	48.9	Zn^{65}	250 days	1.12
	68	18.6	Zn^{69}	14 hours	0.44
Zirconium	94	17.4	Zr^{95}	65 days	0.92
Molybdenum	98	23.8	Mo^{99}	67 hours	0.73
Tantalum	181	100.0	Ta^{182}	111 days	1.2
Tungsten	186	28.7	W^{187}	24 hours	0.76
Beryllium	9	100.0	Be^{10}	2.7 × 10^{6} years	...
Sodium	23	100.0	Na^{24}	15 hours	3.7
Magnesium	26	11.1	Mg^{27}	9.6 minutes	1.01
Aluminum	27	100.0	Al^{28}	2.3 minutes	1.78

Induced Radioactivity:

Table IV lists a few common elements

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[3] As reported by various authorities; see references, p. 216.

or isotopes likely to be found in, or considered for, reactor design. This list is incomplete but serves to show the wide range of radioactivity that can be induced in the materials listed and the variation in half-life to be expected with these induced radioactivities. It is apparent that any prolonged induced radioactivity in magnesium or aluminum alloys would be more a function of the alloying elements used than of the principal material. Radiation damage to aluminum and magnesium is relatively slight, and it is readily apparent that a favorite alloy system in aluminum would be an alloy with magnesium. The extremely short half-life of induced radioactivity in aluminum and magnesium (measured in minutes) contributed to the decision to use aluminum as a principal structural material of all early nuclear reactors. The rapid decay of induced radioactivity in aluminum and magnesium facilitates the transport, repair, or examination of items which have been irradiated, since one must first remove surface contaminants by washing or other means, and then set the part aside to "cool" preparatory to further handling.

Decomposition Products:

In addition to induced radioactivity, certain alloy systems may be undesirable in themselves because of decomposition products expected from prolonged exposure to high levels of radioactivity. The appearance of such materials as mercury, silicon, or sulfur, or possibly lead or bismuth, could contribute to the eventual decomposition of an alloy or the failure of a part. For this reason certain materials or alloying elements likely to give decomposition products of this nature are to be avoided.

Radiation Effects on Mechanical Properties:

Radiation effects on mechanical properties of aluminum alloys most likely to be used in reactor design parallel the work hardening effects of cold working. Yield strength is increased, with only a nominal loss of ductility or increase in notch sensitivity. Conceivably, an aluminum vessel of welded construction could be "radiation treated" to improve its mechanical properties. These effects can be reversed by annealing. Magnesium is similarly affected, but because of its higher notch sensitivity it is conceivable that a stressed magnesium design could become a problem.

NUCLEAR RESEARCH AND SPECIAL REACTORS

It appears at the present time that the greatest applications for aluminum and magnesium will be in the building of research, training, and other special reactors. Research reactors could, of course, include almost any type and any size. As used here, the term would include those relatively low power installations being designed and fabricated for educational institutions and various government and private research facilities to broaden both our knowledge of reactors themselves and our knowledge of the reactions of various materials to radioactive environments with or without simultaneous conditions of high temperatures and pressures. A number of these reactors have been built especially for materials testing, and it appears that a large number of such reactors will be fabricated to provide low-power installations for educational institutions concerned more with the teaching of reactor technology than with the testing of materials or the generation of power. The term "special reactors" may include those designed for the irradiation of foodstuffs as well as those which provide heat, radioactivity, or other environment or combination of environments deemed desirable in conjunction with special chemical processing problems. The light metals may be widely used in these lighter types of reactors because of their ease of fabrication, light weight, freedom from radiation poisoning, good appearance, and similar reasons. At the present time our nuclear requirements, exclusive of internal or integral components exposed to both coolant and high radiation fields, will be fabricated from standard commercial alloys; however, the development of new alloys providing higher strength at elevated temperatures or other desirable properties may change this picture.

SUMMARY

We can be certain that the producers and fabricators of the light metals will be ever vigilant to find applications for these metals. Their advantage of light weight, good appearance, workability, thermal conductivity, low thermal neutron cross-section, and freedom from radiation poisoning can be depended upon to contribute to the ultimate goal of all American industry—to manufacture a superior product at an acceptable price.

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REPORT OF COMMITTEE B-8

ON

ELECTRODEPOSITED METALLIC COATINGS*

Committee B-8 on Electrodeposited Metallic Coatings held two meetings during the year: on October 18, 1957, at the Benjamin Franklin Hotel, Philadelphia, Pa., and on February 13, 1958, at the Hotel Statler, St. Louis, Mo.

The membership of the committee totals 171, of whom 67 are classified as producers, 26 as consumers, 77 as general interest and 15 as consulting members.

The officers elected for the ensuing term of two years are as follows:

Chairman, C. H. Sample.
Vice-Chairman, R. B. Saltonstall.
Secretary, D. M. Bigge.
Advisory Committee Member-at-Large, William Geissman.

The committee is appending to its report a "Summary of Observations of Factors Influencing Adhesion of Organic Coatings to Chromium Plated Surfaces"[1] prepared by Section F, Subcommittee V.

NEW TENTATIVES

The committee recommends for publication as tentative the Recommended Practice for Cleaning Metals Prior to Electroplating, as appended hereto.[2]

REVISION OF TENTATIVE

The committee recommends revisions as follows of the Tentative Specification for Electrodeposited Coatings of Nickel and Chromium on Steel (**A 166 - 55 T**)[3] and continuation of the specification as tentative.

Table I.—Add a Note to Table I to read as follows:

> NOTE.—The thicknesses given in this table are based on tests and experience with composite coatings in which matte nickel was deposited from a Watts type bath and buffed prior to conventional bright chromium plating. If these conditions are altered, different thicknesses may be required to give equivalent performance.

ADOPTION OF TENTATIVE AS STANDARD WITHOUT REVISION

The committee recommends that the Recommended Practice for the Preparation of Copper and Copper-Base Alloys for Electroplating (**B 281 - 53 T**)[3] be approved for reference to letter ballot of the Society for adoption as standard without revision.

REVISION OF STANDARDS, IMMEDIATE ADOPTION

The committee recommends for immediate adoption revisions as indicated of the following standards, and accordingly asks for the necessary nine-tenths affirmative vote at the Annual Meeting in order that the revisions may be referred to letter ballot of the Society:

Standard Specifications for Electrodeposited Coatings on Copper and Copper Base Alloys (B 141 - 55):[3]

Standard Specifications for Electrodeposited Coatings of Nickel and

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* Sixty-first Annual Meeting of the Society, June 22–27, 1958.

[1] See p. 221.

[2] The new tentative appears in the 1958 Book of ASTM Standards, Part 2.

[3] 1955 Book of ASTM Standards, Part 2.

Chromium on Zinc and Zinc Base Alloys (B 142 – 55):[3]

Section 3.—Add a Note to the table of coating thickness, to read as indicated above for Specification A 166 – 55 T.

Standard Methods of Test for Local Thickness of Electrodeposited Coatings (A 219 – 54):[3] The following recommendations are made jointly with Committee A-5 on Corrosion of Iron and Steel:

Section 4.—Revise to read as follows:

4. Several alternative tests are in common use and may be employed by agreement between the vendor and the purchaser. These include dropping and magnetic tests described in Appendices I and II, as well as others involving the use of magnetic or electromagnetic instruments, chemical or electrochemical stripping tests, or (for heavy deposits) micrometers.

TABLE I.—TIME REQUIRED FOR 0.00001 IN. (0.25 μ) OF CHROMIUM ON STAINLESS STEEL IN SPOT TEST USING SOLUTION CONTAINING 20 g PER LITER ANTIMONY TRIOXIDE IN HCl (SP GR 1.16).

(New Table II, Specification A 219 – 54)

Temperature		Time, sec
deg Fahr	deg Cent	
70	21.1......	7.0
75	23.9......	6.0
80	26.6......	5.0
85	29.4......	4.0
90	32.2......	3.5

NOTE.—Tables I and II are applicable when chromium plate is deposited from the conventional sulfate-chromic acid solution under conventional operating conditions. If these procedures are varied it may be necessary to recalibrate the test.

Section 7.—Add to the Note at the end of the section the following: "CAUTION: Spot test results may be erratic when the chromium is deposited directly on copper."

Add a new Paragraph (*c*) to read as follows:

(*c*) *Test for Thickness of Chromium on Stainless Steel.*—The test solution contains 20 g per liter of antimony trioxide in HCl (sp gr 1.16 ± 0.001 at 60 F). If the basis metal is 18-8 stainless steel, gassing will stop when the endpoint is reached. If the basis metal is 17 per cent chromium stainless steel, the rate of gas liberation will change when the endpoint is reached. Gas will continue to be liberated until the acid is spent or the chromium deposit is dissolved. Results are as follows: no black film, no chromium deposit; black film forms but little or no gassing, chromium thickness estimated to be 0.000003 in. or less.

New Table.—Add a new Table II as shown in the accompanying Table I, numbering the present table in Section 8 as Table I.

TENTATIVES CONTINUED WITHOUT REVISION

The committee recommends the continuation without revision of the following tentatives:

Tentative Specification for:

Electrodeposited Coatings of Lead on Steel (**B 200 – 55 T**), and

Chromate Finishes on Electrodeposited Zinc, Hot Dipped Galvanized, and Zinc Die-Cast Surfaces (**B 201 – 55 T**).

EDITORIAL CHANGES

The committee recommends editorial changes as indicated in the following standards and tentatives:

Standard Specifications for Electrodeposited Coatings of Cadmium on Steel (A 165 – 55):[3]

Section 1.—Add a new Note 3 to read as follows:

NOTE 3: *Toxicity.*—Cadmium is toxic and should not be used as a coating for articles used as food or beverage containers, cooking utensils, or articles that may come in contact with food or beverages.

Because of the toxicity of cadmium vapors and cadmium oxide fumes, cadmium-plated articles must not be welded, spot welded, soldered, or otherwise strongly heated without adequate ventilation which will efficiently remove all toxic fumes.

Tentative Specifications for Electrodeposited Coatings of Nickel and Chromium on Steel (A 166 - 55 T):[3]

Tentative Specifications for Electrodeposited Coatings of Nickel and Chromium on Zinc and Zinc-Base Alloys (B 142 - 55):[3]

Tentative Specifications for Electrodeposited Coatings of Lead on Steel (B 200 - 55 T):[3]

Tentative Specifications for Chromate Finishes on Electrodeposited Zinc, Hot-Dipped Galvanized, and Zinc Die-Cast Surfaces (B 201 - 55 T):[3]

Footnotes.—Revise Footnote 4 concerning the salt spray test in Specifications A 166 - 55 T, B 142 - 55, and B 201 - T, and Footnote 5 in Specification B 200 - 55 T to read as follows:

> Committee B-8 has found that different salt spray cabinets operated in accordance with ASTM Method B 117 - 49 T failed to give reproducible results on replicate panels (see *Proceedings*, Am. Soc. Testing Mats., Vol. 54, p. 299 (1954)). Therefore, periods of continuous exposure to the salt spray are not specified herein.

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[4]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Papers, Specifications and Definitions (F. Ogburn, chairman).—Section B, now under chairmanship of William Safranek, is making good progress in preparing a glossary of terms used in connection with electrodeposition.

Section D (D. S. Carr, chairman) is preparing a specification on industrial nickel and chromium coatings.

Section F (Edward Saubestre, chairman) was established to continue the investigation of marking coatings to indicate compliance with ASTM specifications.

Subcommittee II on Performance Tests (W. L. Pinner, chairman).—Section A (D. M. Bigge, chairman) will determine coating thicknesses on Program 4 panels placed on exposure at Kure Beach, N. C., on April 28, 1956 and at Detroit, Mich., on May 1, 1956. These determinations will be made following inspection during May, 1958, in order to permit proper evaluation of performance data.

Section C (R. E. Harr, chairman) placed cadmium-plated panels on exposure at Kure Beach, N. C., New York City, and Rock Island, Ill. on August 5, 1957.

Section F (J. W. Kerstetter, chairman) has approval of a program for exposure testing of wrought and cast aluminum alloy panels plated with copper-nickel-chromium.

Subcommittee III on Conformance Tests (R. F. Ledford, chairman).—Section A (A. H. DuRose, chairman) has studied data obtained by magnetic gage, drop test, and microscopic methods of measuring thickness. ASTM specifications list the microscopic methods as the primary method. The result of the work of Section A thus far indicates that the microscopic method should not be considered as the primary method since the stripping method is less subject to error and, therefore more reproducible. Several members related similar experiences. The Kocour, Dermitron, and Boonton instruments are now being investigated. Paul Croly was appointed Chairman of Section G on Ductility.

Subcommittee IV on Electroplating Practice (Max Frager, chairman).—Section N, under the chairmanship of B. Knapp, was established to prepare a recommended practice on double nickel coatings.

Section K (Sam Spring, chairman)

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[4] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

prepared the proposed recommended practice for cleaning metals prior to electroplating referred to earlier in this report.

Subcommittee V on Supplementary Treatments (G. E. Best, chairman).—G. E. Best replaces Marc Darrin, who served faithfully for several years as chairman of Subcommittee V.

A new section is planned to consider writing specifications for supplementary coatings on aluminum, magnesium, and their alloys. Cross-representation with Committee B-7 on Light Metals and Alloys is planned.

This report has been submitted to letter ballot of the committee, which consists of 171 voting members: 94 members returned their ballots, of whom 93 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

C. H. SAMPLE,
Chairman.

D. M. BIGGE,
Secretary.

APPENDIX

SUMMARY OF OBSERVATIONS OF FACTORS INFLUENCING ADHESION OF ORGANIC COATINGS TO CHROMIUM PLATED SURFACES

First Progress Report of Section F on Organic Finishes on Plated Metal Coatings, of Subcommittee V on Supplementary Treatments for Metallic Coatings

This report summarizes the observations that have been made regarding factors which influence the adhesion and retention of adhesion of (clear) organic coatings to chromium plate. The purpose of this report is to serve as a guide for those interested in the use of supplementary organic coatings on chromium-plated surfaces.

In the observations reported below, several methods of determining adhesion were used. In general, initial adhesion was determined by scribing through the organic coating to the plated surface and measuring adhesion by either the knife test or the tape test. Retention of adhesion was determined after exposing the coated chromium-plated articles to outdoor weathering, to humidity, to water immersion, or to salt spray, after which the coating was scribed to the plated surface and the adhesion determined by the knife test or the tape test.

Summary of Observations

As on any metal surface, the specific formulation of the organic coating and its application to the plated surface are important factors influencing adhesion to the chromium plate. However, these factors are not within the scope of this study. Furthermore, this study is not concerned with such properties of the organic coating as exterior durability, resistance to discoloration, resistance to humidity, hardness, and marring, although these properties are important depending upon the specific service requirements of the finished article.

In the following comments, it is assumed that the organic coating under consideration does have good adhesion to chromium plate that has been plated under ideal conditions and that the application of the coating is accomplished in such a manner as to develop maximum properties for the service intended, that is, applied at the proper thickness to form a continuous film, properly dried and so on.

Ideal Conditions:

The organic coating is applied to a plated surface deposited from a homogeneous chromium plating solution in which the final rinses are distilled or demineralized water and the organic coating is applied immediately after the plated surface is dried. Under these conditions, optimum adhesion and retention of adhesion under exposure conditions is obtained.

Variations from the Ideal Which Affect Adhesion:

1. The time interval between plating and coating is important. If the plated parts are exposed to plating room atmosphere, that is, humid, acidic, or alkaline atmosphere, this time interval must be short. General observations indicate that longer than 2 to 4 hr exposure to plating room atmosphere is detrimental to adhesion. If the plated parts are

exposed to clean and dry atmosphere such as may exist in a coating department area, the time factor is not critical. Two days may elapse under such conditions without affecting adhesion.

2. Hardness of the Final Rinse Waters.—Poor adhesion has been reported with as little as 2 ppm in the final rinses. With chromic acid treatment of the chromium plate (described below), good adhesion has been reported with as high as 170 ppm.

3. Irregular Additions of Chemicals for Chromium Plating Bath Maintenance.—Poor adhesion has been reported immediately following large additions of chromic or sulfuric acid or water for makeup purposes. However, this has not been completely confirmed, especially when the plating solution is mixed until homogeneous after such additions. It is suggested that irregular additions may be a cause of poor adhesion if no other factors for the poor adhesion are apparent.

4. No effect on adhesion has been observed with variation in the chromic acid to sulfate ratio or in current densities in the range normally employed.

5. Differences in the base metal (steel, zinc, or brass) do not affect adhesion on copper-nickel-chromium plate.

6. Variations in nickel plate thickness do not appear to affect adhesion. A flash of nickel is equal to 0.0005 in. of nickel.

7. Adhesion is poorer on chromium plated directly on copper without nickel than on chromium plated over nickel.

Effects of Various Treatments of the Chromium Plated Surface to Overcome Variations from the Ideal:

1. Sulfuric acid treatment causes poor adhesion. A quick dip in hydrochloric acid results in poor adhesion. Contradictory results have been reported with phosphoric acid treatment.

2. Vapor degreasing does not improve adhesion.

3. Dilute chromic acid treatment is the most effective. This consists of immersing or spraying with a solution consisting of 2 to 4 oz of chromium oxide per 100 gal of demineralized or distilled water. Variables in the treatment affect adhesion as follows:

(*a*) Temperature variations between 140 and 180 F do not influence results.

(*b*) Variations in treating time between 10 sec and 2 min have no effect. Both treating times are satisfactory.

(*c*) Low solution temperatures such as 110 F are reported to be less effective than 140 F.

(*d*) Sulfate contamination of the solution, which may occur if plating solution is used to prepare the dilute chromic acid solution or if dragout occurs, requires longer treating time; 2 min is more effective than 10 sec.

(*e*) The final rinse after treatment with dilute chromic acid is important. Demineralized water should be employed. If the final water rinse has a pH above 7, poor adhesion will result. Without a rinse, visible chromic acid stains occasionally occur; such stains result in poor adhesion.

(*f*) The time interval between treatment and coating is important. Shorter times produce the best results, as noted above.

On Buffed Chromium Plate:

1. Vapor degreasing, washing with hydrocarbon solvent or alcohol, or alcohol containing traces (100 ppm) of chromium oxide are not effective in obtaining adhesion.

2. Electrocleaner followed by dilute chromic acid treatment as described above is necessary to obtain adhesion.

Respectfully submitted on behalf of Section F of Subcommittee V,

F. L. SCOTT,
Chairman.

REPORT OF COMMITTEE B-9

ON

METAL POWDERS AND METAL POWDER PRODUCTS*

Committee B-9 on Metal Powders and Metal Powder Products held one meeting during the year: in New York City on February 21, 1958.

The officers and members-at-large of the Advisory Committee elected for the ensuing term of two years are as follows:

Chairman, J. L. Bonanno.

Vice-Chairman, H. R. Biehl.

Secretary, C. G. Johnson.

Advisory Committee Members-at-Large: G. L. Bibbins, H. B. Huntress, F. V. Lenel, and G. L. Werley.

NEW TENTATIVES

The committee recommends for publication as tentative the following methods as appended hereto:[1]

Tentative Methods of Test for:

Density and Interconnected Porosity of Sintered Powder Metal Structural Parts and Oil-Impregnated Bearings,

Average Particle Size of Refractory Metals and Compounds by Fisher Subsieve Sizer,

Apparent Density of Refractory Metals and Compounds by Means of the Scott Volumeter, and

Determination of the Compressibility of Metal Powders.

REVISIONS OF TENTATIVES

The committee recommends revisions as indicated of the following tentatives and their continuation as tentative:

Tentative Specifications for Metal Powder Sintered Bearings (Oil Impregnated) (B 202 - 55 T):[2]

Section 5.—Replace the present Paragraphs (*b*) and (*c*) with the following new Paragraph (*b*):

> (*b*) Density shall be determined in accordance with the Methods of Test for Density and Interconnected Porosity of Sintered Powder Metal Structural Parts and Oil-Impregnated Bearings (ASTM Designation: B 328."

Section 6.—Delete the entire section and replace with the following: "6. Porosity in terms of interconnected void space shall not be less than 18 per cent for all classes when determined in accordance with ASTM Method B 328."

Tentative Specifications for Sintered Metal Powder Structural Parts from Bronze (B 255 - 54 T):[2]

Section 5 (*c*).—Revise to read as indicated above for new Paragraph (*b*) in Section 5 of Specifications B 202.

Section 6 (*b*).—Delete the entire Paragraph (*b*) and replace with "(*b*) The porosity shall be determined in accordance with ASTM Method B 328."

Section 7.—Change the heading to read "Mechanical Properties." In the third line and in the Note change the words "physical properties" to read "mechanical properties."

Section 8.—Change the last sentence of Paragraph (*b*) to read: "In order to obtain oil-free chips, the pieces selected for test shall have, if necessary, the oil extracted in accordance with ASTM Method B 328."

In the heading and in the last line of Paragraph (*c*) change "physical" to read "mechanical."

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* Sixty-first Annual Meeting of the Society, June 22-27, 1958.

[1] The new tentatives appear in the 1958 Book of ASTM Standards, Part 3.

[2] 1955 Book of ASTM Standards, Part 2.

Section 12.—Change to read "12. Unless otherwise specified, rejection based on tests made in accordance with these specifications shall be reported to the manufacturer."

Appendix.—In Paragraph A1 delete the last sentence.

Tentative Specifications for Sintered Metal Powder Structural Parts from Brass (B 282 – 53 T):[2]

Section 5 (c).—Delete this paragraph and replace with the following:

(*c*) Density measurements shall be made in accordance with the Method of Test for Density and Porosity of Sintered Powder Metal Structural Parts and Oil-Impregnated Bearings (ASTM Designation: B 328).

Section 6 (b).—Delete this paragraph and the Note and replace with the following:

(*b*) Porosity of class A brass shall be calculated in accordance with Method B 328.

Section 7 (a).—In the heading, the third line, and in the Note change "physical properties" to read "mechanical properties." Before the last sentence insert a new sentence to read "Hardness on specified areas of the part can be determined by either the conventional Brinell hardness test or by the Rockwell hardness test employing the H scale (⅛-in. diameter ball, 60-kg load)."

Section 8.—Change the last sentence of Paragraph (*b*) to read: "In order to obtain oil-free chips, the parts selected for test shall have the oil extracted in accordance with ASTM Method B 328, if necessary."

In the heading and the fifth line of Paragraph (*c*) change "physical tests" to read "mechanical tests."

Section 12.—Replace with "12. Unless otherwise specified, rejection based on tests made in accordance with these specifications shall be reported to the manufacturer."

Appendix.—Delete the Appendix and replace with the following, adding a new Table II as shown in the accompanying Table I:

A1. Data for the mechanical properties of sintered brass molded specimens are given in Table II. The data do not constitute a part of the specifications. They merely indicate to the purchaser the mechanical properties of which these materials are capable from special subsize tension and compression specimens conforming to the density and chemical composition requirements specified. The tensile values represent specimens molded to the size specified for the ASTM unmachined flat tension bar and the machined round tension bar. The compression tests are run on molded specimens with a diameter of ½ in. and a length of ¾ in. The Rockwell H scale hardness readings were taken on the grip ends of ASTM unmachined flat tension bars in the as-sintered condition.

TABLE I.—TYPICAL MECHANICAL PROPERTIES.
(New Table II, Specification B 282)

Density, g per cu cm	Ultimate Tensile Strength, psi	Elongation in 1 in., per cent	Rockwell Hardness, H scale	Yield Point in Compression, 0.1 per cent offset, psi
7.2	20 000	9.0	64	10 000
7.7	23 000	10.0	70	12 000
8.0	27 000	13.0	85	14 000

A2. Hardness values, as determined by conventional Brinell or Rockwell H scale tests, have been found useful in product control and afford relatively good correlation with density and tensile strength if they are determined on parts in the as-sintered condition. For parts used in the as-sintered condition or after having been sized only very lightly, hardness may therefore be used as a qualification test as outlined in Section 7(*a*). In this case, the specific area or areas of the part on which the hardness requirements must be met shall be carefully specified.

Tentative Specifications for Copper-Infiltrated Iron Parts (B 303 – 55 T):[2]

Section 5 (c).—Replace this paragraph with the following:

(*c*) The density shall be determined in the same manner as porous materials to avoid errors that may occur in parts which may not be completely copper infiltrated. It shall be determined in accordance with the Methods of Test for Density and Interconnected Porosity of Sintered Powder Metal Structural Parts and Oil-Impregnated Bearings (ASTM Designation: B 328).

Section 6.—In the heading and in the third line change "physical properties" to read "mechanical properties."

Section 7.—Change the last sentence of Paragraph (*b*) to read: "In order to obtain oil-free chips, the parts selected for test shall have the oil extracted in accordance with ASTM Method B 328, if necessary."

In the heading and in the fifth line of Paragraph (*c*), change "physical tests" to read "mechanical tests."

Section 11.—Change to read "11. Unless otherwise specified, rejection based on tests made in accordance with these specifications shall be reported to the manufacturer."

Appendix.—In the sixth line, change "physical properties" to read "mechanical properties."

Tentative Specifications for High Density Iron, Sintered Metal Powder Structural Parts (B 309 – 56 T):[3]

Section 5 (c).—Change to read as indicated above for new Paragraph (*b*) in Section 5 of Specifications B 202.

Section 7 (b).—Change the last sentence to read: "In order to obtain oil-free chips, the parts selected for test shall have the oil extracted in accordance with ASTM Method B 328, if necessary."

Section 11.—Change to read: "11. Unless otherwise specified, rejection based on tests made in accordance with these specifications shall be reported to the manufacturer."

Tentative Specifications for Low and Medium Density Iron, Sintered Metal Powder Structural Parts (B 310 – 56 T):[3]

Section 5 (c).—Change to read as indicated above for new Paragraph (*b*) in Section 5 of Specifications B 202.

Section 6 (b).—Delete this paragraph and the Note following it and replace with "(*b*) The porosity shall be determined by ASTM Method B 328."

Section 8 (b).—Change the last sentence to read: "In order to obtain oil-free chips, the parts selected for test shall have the oil extracted in accordance with ASTM Method B 328, if necessary."

Tentative Method of Test for Green Strength of Compacted Metal Powder Specimens (B 312 – 56 T):[3]

Section 3 (a).—Change to read as follows:

> 3. (*a*) Lubrication is performed by rinsing the die wall with the lubricant (a mixture of 100 g of zinc stearate in 1 liter of methyl chloroform, 1,1,1-trichloroethane). Immediately drain the die, dry, and fill with the powder to be tested, as described in Paragraphs (*b*) and (*c*).

ADOPTION OF TENTATIVE AS STANDARD WITHOUT REVISION

The committee recommends that the Tentative Method of Test for Density of Cemented Carbides (**B 311 – 56 T**)[3] be approved for reference to letter ballot of the Society for adoption as standard without revision.

REVISIONS OF STANDARD, IMMEDIATE ADOPTION

The committee recommends for immediate adoption revisions as indicated of the following standards, and accordingly asks for the necessary nine-tenths affirmative vote at the Annual Meeting in order that the revisions may be referred to letter ballot of the Society:

Standard Specifications for Sintered Metal Powder Structural Parts (B 222 – 52):[2]

Section 5 (c).—Change to read as

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[3] 1956 Supplement to Book of ASTM Standards, Part 2.

indicated above for new Paragraph (*b*) in Section 5 of Tentative Specifications B 202.

Section 6 (b).—Change to read: "The porosity shall be determined by ASTM Method B 328."

Section 7 (a).—In the heading, the third line, and in the Note change "physical properties" to read "mechanical properties."

Section 8.—Change the last sentence of Paragraph (*b*) to read: "In order to obtain oil-free chips, the parts selected for test shall have the oil extracted in accordance with ASTM Method B 328, if necessary."

In the heading and in the last line of Paragraph (*c*), change "physical tests" to read "mechanical tests."

Section 12.—Change to read: "Unless otherwise specified, rejection based on tests made in accordance with these specifications shall be reported to the manufacturer."

Appendix.—Delete the last sentence.

Standard Definitions of Terms Used in Powder Metallurgy (B 243 – 55):[2] Add the following new definitions:

K-Factor.—The strength constant in the formula for Radial Crushing Strength of a plain sleeve specimen of sintered metal. See also Radial Crushing Strength; see also ASTM Specifications B 202.

Radial Crushing Strength.—The relative capacity of a plain sleeve specimen of sintered metal to resist fracture induced by a load applied between flat parallel plates in a direction perpendicular to the axis of the specimen. See ASTM Specifications B 202.

Infiltration.—A process of filling the pores of a sintered, or unsintered, compact with a metal or alloy of lower melting point.

STANDARDS CONTINUED WITHOUT REVISION

The committee recommends the continuation without revision of the following standards:

Standard Method of:

Test for Apparent Density of Metal Powders (**B 212 – 48**),
Test for Flow Rate of Metal Powders (**B 213 – 48**), and
Sampling Finished Lots of Metal Powders (**B 215 – 48**).

TENTATIVES CONTINUED WITHOUT REVISION

The committee recommends the continuation without revision of the following tentatives:

Tentative Method for:

Subsieve Analysis of Granular Metal Powders by Air Classification (**B 293 – 54 T**).

Tentative Recommended Practice for:

Hardness Testing of Cemented Carbides (**B 294 – 54 T**).

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[4]

ADVISORY COMMITTEE ACTIVITIES

The Advisory Committee held two meetings during the year. A number of committee personnel changes were approved. Mr. W. Babington was appointed to represent Committee B-9 on Committee E-11 on Quality Control of Materials. Mr. John Redmond was appointed Chairman of III-C upon the resignation of Mr. Engle. An Editorial Subcommittee consisting of Mr. Babington, Chairman, M. Bozsin, and Fritz V. Lenel was formed. This subcommittee is charged with editing all specifications before their submission to the Society for publication. It was decided to publish the specifications under the jurisdiction of Committee B-9 in a separate bound volume. Harold Biehl was asked to prepare an article on B-9 research activities for the 1958 ASTM publication on research. Committee B-9, through the efforts of M. H. Meighan, is aiding the Society of Automotive Engineers in the preparation of information and specifications for the SAE Handbook. Further expansion of the work of Committee B-9 into various facets of industry where powder metallurgy plays a part is being explored. Subcommittee III-A will consider the field of steelbacked bearings.

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[4] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

ACTIVITIES OF SUBCOMMITTEES

The eight active subcommittees and sections of the committee met twice during the year; in Chicago, Ill. on November, 1957, and prior to the main committee meeting in New York City on February 20 and 21, 1958.

Subcommittee I on Nomenclature and Technical Data (F. N. Rhines, chairman) revised the present glossary (B 243 – 55), adding three terms and definitions referred to earlier in this report. Still under consideration is a name for the end-product of the powder metallurgy industry. Action is continuing on the analysis of technical data as it affects the value of specifications such as B 222 – 52, and it is expected work will continue on a broader basis than heretofore.

Subcommittee II on Metal Powders (J. B. Haertlein, chairman):

Section A on Base Metal Powders (J. B. Haertlein, chairman) drafted the proposed Tentative Method of Test for Determination of Compressibility of Metal Powders, which is being submitted to the Society for publication. A study is being made of furnaces and controls necessary in order to standardize methods for the sintered properties of metal powders. The need for compressibility data in the high-pressure range has prompted this committee to appoint a task group charged with the duty to investigate and gather data on this problem.

Section B on Refractory Metal Powders (W. H. Bleecker, chairman) prepared and submitted for publication by the Society the Method of Test for Average Particle Size of Refractory Metals and Compounds by Fisher Subsieve Sizer and the Method of Test for Apparent Density of Refractory Metals and Compounds by Means of the Scott Volumeter referred to earlier in this report. A task force working with Refractory Group 2 of Committee E-3 is working on a procedure for analysis of nonvolatile matter. The subcommittee will continue work on the turbidimetric method and on microscopic techniques for particle size.

Subcommittee III on Metal Powder Products (R. P. Koehring, chairman):

Section A on Bearings (M. H. Meighan, chairman) voted the retention as tentative of the Specifications for Metal Powder Sintered Bearings (Oil Impregnated) (B 202 – 55 T) with the revisions referred to earlier in this report. Correlation of *K*-factor, microstructure, and machinability of sintered bronze bearings is progressing satisfactorily. A proposed size list for flanged sleeve-type bearings and for washers has been prepared and will be submitted to the subcommittee for letter ballot. A task group is working to submit more definite limits for copper content of type II class B material than the present 5 to 30 per cent copper range contained in Specification B 202 – 55 T. This committee will carry on research into the need for specifications in the field of sintered bronze on steel bushings and washers.

Section B on Structural Parts (P. J. Shipe, chairman) prepared the Method of Test for Density and Interconnected Porosity of Sintered Powder Metal Structural Parts and Oil-Impregnated

Bearings referred to earlier in the report. This committee plans to draft an introductory educational preface for specifications. Upon evaluation of questionnaires regarding mechanical data it was decided there was no need to upgrade mechanical properties data at this time. A program is under way to expand specifications covering high-density iron parts. Also, a study is being made of surface finish of sinterings and methods of measuring surface finish of metal powder products.

Section C on Cemented Carbides (J. C. Redmond, chairman) is drafting an introductory educational preface for specifications. Upon evaluation of questionnaires relative to mechanical properties data contained in specifications prepared by this subcommittee, it was decided there was no need to upgrade or change these data at present. Also, a study is being made of surface finish and methods of measuring surface finish of metal powder products.

Three task groups have been appointed to study hardness testing, physical properties, and metallographic characteristics of carbides.

Section D on Friction Materials (H. B. Huntress, chairman) has developed specifications for hardness, density, and transverse rupture strength. A second round of friction testing using standard specimens is being carried out with the objective of writing a standard procedure for testing for friction properties of sintered metal friction materials. Specific heat, thermal conductivity, melting point, and corrosivity are all facets relative to preparing specifications which will require evaluation by this subcommittee.

Section E on Machinable Heavy Metals (A. S. Doty, chairman) has prepared for publication a specification for machinable high-density tungsten-nickel-copper alloys which is now being reviewed by the Editorial Committee.

This report has been submitted to a letter ballot of the committee, which consists of 79 members; 56 members returned their ballots, of whom 53 have voted affirmatively and 1 negatively.

Respectfully submitted on behalf of the committee,

J. L. BONANNO,
Chairman.

C. G. JOHNSON,
Secretary.

REPORT OF ADVISORY COMMITTEE

ON

CORROSION

The Advisory Committee held two meetings during the year: on June 20, 1957 at Atlantic City, N. J., and November 25, 1957, at ASTM Headquarters, Philadelphia, Pa.

Personnel

Committee D-15 on Engine Antifreezes has asked to be represented on the Advisory Committee and has appointed R. E. Vogel and C. H. Sweatt as representatives. New committee representatives are as follows: for Committee A-3, T. E. Eagan replaces D. E. Krause; for Committee A-5, H. F. Hormann replaces Marc Darrin; for Committee A-7, W. M. Albrecht succeeds W. A. Kennedy; for Committee A-10, L. L. Wyman replaces F. L. LaQue; for Committee C-19, T. P. Pajak replaces George Gerard; for Committee D-14, J. E. Rutzler replaces R. F. Blomquist; for Committee D-19, F. L. LaQue succeeds F. N. Alquist; and, for Committee D-20, J. H. Gibbud is the alternative member rather than H. A. Perry as given in the 1957 report. Committee B-4 has discontinued its representation.

Mr. C. P. Larrabee has been recommended for reappointment as a member-at-large for a three-year term from June, 1958.

Status of Atmospheric Exposure Programs

Committee A-5 on Corrosion of Iron and Steel:

A new program on testing hardware, which will supplement the earlier program begun 28 years ago and still continuing at the State College (Pa.) Test Site, will be started this year. Approximately 750 flat, bent, and round specimens made of carbon steel, four low-alloy steels, and two nodular irons in the bare condition and coated with hot-dipped, sprayed, and electrodeposited zinc, and hot-dipped and sprayed aluminum will be tested. These specimens will be exposed at Newark, N. J., and at the 80- and 800-ft test sites at Kure Beach, N. C. Data to be obtained from the exposure will include pit depth and time to coating failure over a period of 20 years.

Committee A-7 on Malleable Iron Castings:

A series of 1620 malleable iron plates are being exposed at the test sites located at Point Reyes, Calif., East Chicago, Ind., New York Area (Newark, N. J.), Kure Beach, N. C., and State College, Pa. This program includes 600 standard malleable, 600 pearlitic malleable, 240 nodular iron, and 180 steel specimens. Half of the cast plates will be exposed with completely machined surfaces, half with as-cast surfaces. The pearlitic malleable plates will be of both low and high combined carbon types. One group of nodular plates is as-cast, the other heat-treated. The wrought plates are of structural carbon copper-bearing, and low-alloy steels. Specimens will be measured for loss in weight and will be on exposure for periods of 1, 3, 7, and 12 years.

Committee A-10 on Iron-Chromium, Iron-Chromium-Nickel, and Related Alloys:

A large exposure program to determine the corrosion characteristics of currently produced wrought stainless-steel products will be placed on exposure during 1958 at the New York Area (Newark, N. J.), Columbus, Ohio, State College, Pa., Freeport, Tex., and the 80- and 800-ft test sites at Kure Beach, N. C. Over 5000 stainless steel panels and coiled wire springs were prepared to determine changes in tensile and fatigue strength and appearance over the 15-yr exposure period of the test.

Committee B-3 on Corrosion of Non-Ferrous Metals and Alloys:

A large program is being undertaken which will involve exposure of 60 specimens each of 74 commercially produced alloys to be placed on exposure at State College, Pa., New York Area (Newark, N. J.), the 80-ft site at Kure Beach, N. C., and Point Reyes, Calif. The specimens will be used to determine change in weight and strength due to atmospheric corrosion, and the depth of pits will be measured. The exposure will be for periods of 1, 2, 7, and 20 years.

Specimens of two magnesium alloys coupled to other metals are now being assembled to test a bolted galvanic couple arrangement which is expected to give more definitive results than the disk-type couples. These will be placed on exposure at State College, Pa., New York Area (Newark, N. J.), and the 80- and 800-ft sites at Kure Beach, N. C. for 15 years.

Committee B-7 on Light Metals and Alloys, Cast and Wrought:

The data from 3 years atmospheric exposure of aluminum and magnesium sand and permanent mold castings are published in the 1958 Annual Report of Committee B-7.[1] Specimens exposed for 5 years have been withdrawn and are under evaluation.

Committee B-8 on Electrodeposited Metallic Coatings:

A series of 780 chromated cadmium-plated panels representing 14 test films has been placed on exposure at test sites at Rock Island, Ill., New York Area (Bell Telephone Laboratories, N. Y.), and the 800-ft site at Kure Beach, N. C. This program is designed to establish standards for the evaluation of the additional protection afforded by various types of chromate films. A portion of the test panels is provided with buttons of dissimilar metals for study of galvanic action as influenced by the chromate film.

An inspection of the copper-nickel-chromium and nickel-chromium Program 4 panels was made in 1956 and 1957. These data are being evaluated: to determine the effect of a nickel strike and a copper strike under bright nickel, to determine the effect of chromium thickness over nickel (and, insofar as possible, the effect of residual stress of the chromium), to determine the effect of buffing nickel, and to compare buffed Watts nickel with bright nickel.

Committee C-19 on Structural Sandwich Construction:

Some specimens from the second sandwich panel test program started in 1957 are being removed for examination and testing. The first sandwich panel test program was begun in 1955 and completely removed in 1958 for examination and testing. A report on the data from the first test program is in preparation.

STATUS OF CORROSION TEST METHODS

Committee D-10 on Shipping Containers:

A method to measure the corrosive, staining, or tarnishing characteristics of

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[1] See p. 194.

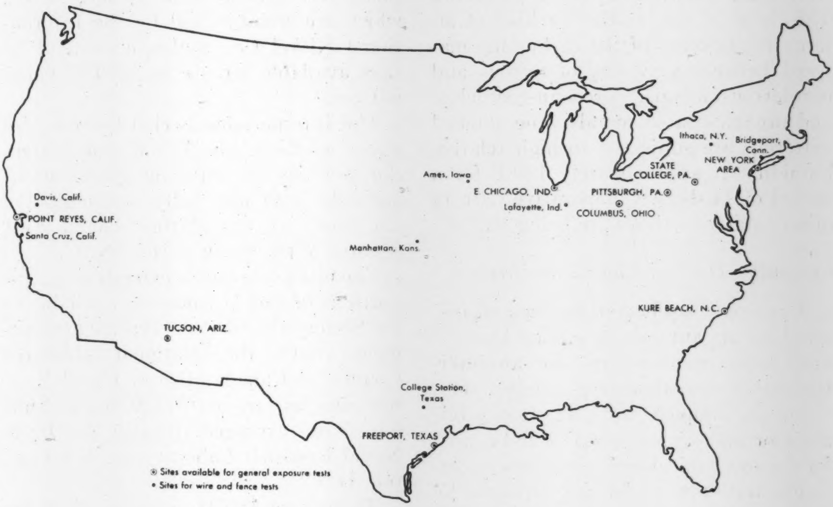
Ithaca, N.Y.
Bridgeport, Conn.
NEW YORK AREA
STATE COLLEGE, PA.
Ames, Iowa
E. CHICAGO, IND.
PITTSBURGH, PA.
Lafayette, Ind.
COLUMBUS, OHIO
Davis, Calif.
POINT REYES, CALIF.
Santa Cruz, Calif.
Manhattan, Kans.
KURE BEACH, N.C.
TUCSON, ARIZ.
College Station, Texas
FREEPORT, TEXAS
⊙ Sites available for general exposure tests
• Sites for wire and fence tests

FIG. 1.—Location of Test Sites Maintained by the Advisory Committee on Corrosion.

TABLE I.—ASTM ATMOSPHERIC EXPOSURE SITES AVAILABLE FOR GENERAL TESTS.

Rural Atmosphere	State College, Pa. (40°48′N–77°52′W)
Urban or Moderate Industrial Atmosphere	Columbus, Ohio (40°00′N–83°01′W)
Industrial Atmosphere	East Chicago, Ind. (41°37′N–87°29′W)
Severe Industrial Atmosphere	Pittsburgh, Pa. (40°27′N–80°01′W)
	New York Area:
	Bell Telephone Laboratories, N. Y. (40°44′N–74°00′W)
	Port Authority Building, N. Y. (40°44′N–74°00′W)
	Newark, N. J. (40°42′N–74°11′W)
West Coast Marine Atmosphere	Point Reyes, Calif. (38°05′N–122°57′W)
East Coast Marine Atmosphere	Kure Beach, N. C., 800-ft site (34°00′N–77°54′W)
East Coast Marine Atmosphere, Beach Exposure	Kure Beach, N. C., 80-ft site (34°00′N–77°54′W)
Gulf Coast Atmosphere	Freeport, Tex. (28°53′N–95°22′W)
Desert Atmosphere	Tucson, Ariz. (32°13′N–111°00′W)

cushioning materials is being proposed. This method entails the stacking of alternate specimens of the cushioning material between a variety of ferrous and non-ferrous metals, porcelain-enameled, and organic-coated metals. The stacked materials are subjected to high relative humidity at approximately 120 F for a period of 14 days. Comparative data of other similar methods are being studied.

Committee D-15 on Engine Antifreezes:

A reservoir equivalent in size and material to an automobile engine block is now being manufactured for an interlaboratory collaborative study. This reservoir, coupled with an automobile radiator and water pump, will be used for the mechanical bench equipment corrosion test now under development by the committee. This method will supplement the Method for Glassware Corrosion Test for Engine Antifreezes (D 1384).

ASTM TEST SITE PROGRAM

Over 50 years have elapsed since the first atmospheric exposure test program was started under the auspices of ASTM. In October, 1906, Committee E on Preservative Coatings for Iron and Steel applied 57 different paints to 2 spans of the Pennsylvania Railroad deck truss bridge at Havre de Grace, Md. Since that time, ASTM committees have utilized some 60 atmospheric test sites, 6 fresh and sea water immersion test sites, and 7 indoor test locations.

Presently, the Advisory Committee, in addition to coordination of corrosion investigations, researches, and test procedures of other committees of the Society, is charged with the responsibility of maintaining 17 test sites whose location in the United States is shown in Fig. 1. Eight of these test sites are for the exclusive use of the Committee A-5 wire and fencing test program. Two additional test sites at Kure Beach, N. C., which are administered by the International Nickel Co., makes a total of 12 sites available for use by ASTM Committees.

The International Nickel Co. has also made available to ASTM committees the facilities for exposing specimens at half-tide level and fully submerged in sea water at the Harbor Island Test Station, Wrightsville Beach, N. C.

Canadian test sites extending as far north as 65 deg latitude are available to the Society through cooperative arrangements with the National Research Council of Canada. Use of Canal Zone test sites as far south as 9 deg latitude have been arranged through the U. S. Naval Research Laboratories, Washington, D. C.

The twelve ASTM test sites which are available for general use by ASTM committees are listed in Table I and information about each follows. The atmospheric exposure test programs which have been carried out at these sites are listed in Table II.

State College, Pa.:

This rural site was established in 1925 and is located one mile north of State College, Pa. (elevation, 1175 ft). The specimens are mounted 30 deg from the horizontal and face SE at an azimuth of 147 deg.

Columbus, Ohio:

This urban or moderate industrial test site was established in 1950 and is located on the roof of the 7th Ave. Building, Battelle Memorial Inst. (elevation, 815 ft). The specimens are mounted at 30 deg from the horizontal and face due south.

East Chicago, Ind.:

This site was established in 1951 as an industrial site typical of the Middle West. Since no calibration studies have

TABLE II.—ATMOSPHERIC EXPOSURE CORROSION TESTS CARRIED OUT AT ASTM TEST SITES BY ASTM COMMITTEES AND OTHER ORGANIZATIONS.

The boldface numbers refer to the table of references given on p. 236.

Material Exposed	Bare Ferrous Metal	Hot-Dip Coatings	Electrodeposited Coatings	Bare Non-Ferrous Metal	Sprayed Coatings	Clad Coatings	Galvanic Couples	Sandwich Constructions
State College, Pa.	A-5 (**1, 2, 3, 4, 5, 6**) B-3 (**9**)	A-5 (**1, 2, 3, 4, 5, 6**)	B-8 (**28, 29, 30, 31, 32, 33**)	B-3 (**9, 11, 12, 13, 14, 15**) B-6 (**20, 21, 22, 23**) B-7 (**24, 25, 26, 27**)	...	B-7 (**24, 25, 27**)	B-3 (**10, 16, 17, 18, 19**)	C-19[e]
Columbus, Ohio	...	...	...	...	AWS[d]	...	...	...
East Chicago, Ind.	SFSA[a]	...	...	...	AWS[d]	...	...	...
Pittsburgh, Pa.	A-5 (**34**) A-10 (**35**)	...	...	...	...	...	...	...
Bell Telephone Labs., N. Y., N. Y.	...	...	A-5 (**7, 8**) B-8 (**28, 29, 30, 31, 32, 33, 40**)	B-3 (**11, 12, 13, 14, 15**) B-6 (**20, 21, 22, 23, 38, 39**)	...	...	B-3 (**10, 16**)	...
Port Authority Bldg., N. Y., N. Y.	A-10 (**35**)	...	...	B-3 (**9, 17, 18, 19**) B-6 (**37**) B-7 (**24, 25, 26, 27**)	...	B-7 (**24, 25, 27**)	...	...
Newark, N. J.	A-10 (**35**)	...	M & T[c]	B-3 (**9**)	AWS[d]	...	...	...
Point Reyes, Calif.	...	...	B-8 (**40**)	B-3 (**9**) B-7 (**24, 25, 26, 27**)	AWS[d]	B-7 (**24, 25, 27**)	...	...
Kure Beach, N. C., 800-ft site	(**36**) INCO[b]	(**36**) INCO[b]	(**36**) INCO[b] B-8 (**30, 40**)	(**36**) INCO[b] B-7 (**24, 25, 26, 27**) B-6 (**37**)	AWS[d]	B-7 (**24, 25, 27**)	(**36**) INCO[b] B-3 (**9, 16, 17, 18, 19**) B-8 (**30, 31, 32, 33**)	...
Kure Beach, N. C., 80-ft site	(**36**) INCO[b]	(**36**) INCO[b]	(**36**) INCO[b] B-8 (**40, 41**)	(**36**) INCO[b]	...	...	(**36**) INCO[b]	C-19[e]
Freeport, Tex.	...	...	...	B-7 (**24, 25, 26, 27**) B-6 (**37**)	AWS[d]	B-7 (**24, 25, 27**)	...	...

[a] Steel Founders Society of America. No specimens have been removed for evaluation as yet.

[b] International Nickel Co. supplied data for this lecture.

[c] Metal & Thermit Corp. No specimens have been removed for evaluation as yet.

[d] American Welding Society. Report on the first set of specimens is in preparation.

[e] Report on sandwich panels exposed at these sites is in preparation by Committee C-19 on Structural Sandwich Constructions.

been conducted at this site, it is assumed to be severe industrial in character. This test site is in an open field bordering the Calmet River 4.2 miles southwest of Lake Michigan (elevation, 600 ft). The specimens are mounted at an angle of 30 deg from the horizontal and face due south.

Pittsburgh, Pa.:

The present severe industrial Pittsburgh test site is only of limited extent and is located on the roof of the Williams and Co. building, Galveston and Pennsylvania Ave., Pittsburgh, Pa. (elevation, 750 ft). This is the fifth site in the Pittsburgh area. This area was originally chosen for its severely corrosive atmosphere high in SO_2. Recent large-scale substitution of natural gas for coal and smoke abatement programs have changed the corrosivity of the atmosphere.

The first test site was located on the grounds of the Carnegie Technical Schools where Committee A-5 mounted wire and fencing in September, 1908. The second site was at Fort Pitt Arsenal, where Committee A-5, in December, 1916, placed a series of corrugated steel and iron sheets. This test was completed in March, 1923. The third test site, on Brunot Island (Duquesne Light Co.), was established in 1926 and was discontinued in 1951. Committees A-5 and B-3 established programs here and in 1951 the Committee A-5 sheet tests were discontinued. The wire tests were transferred to the U. S. Bureau of Mines. The fourth test site was established on the roof of the Bureau of Mines in 1938. Committee A-5 maintained programs at this site and moved the specimens in 1952 to the present location at Williams and Co. Specimens face in two directions, SSE and NNW.

Bell Telephone Laboratories, New York, N. Y.:

Panel specimens face SSE at an azimuth of 154 deg and are located on the roof of the Bell Telephone Laboratories in downtown New York City in an area of severe industrial exposure.

Port Authority Building, New York, N. Y.:

In 1948 an area on the 16th floor roof of the New York Port Authority Building was set aside as a severe industrial site. Since then, a heliport has been constructed adjacent to and 10 ft above it which will reduce its usefulness as a test site. The specimens are mounted at an angle of 30 deg from the horizontal and face SSW at an azimuth of 210 deg behind a 10-ft parapet wall.

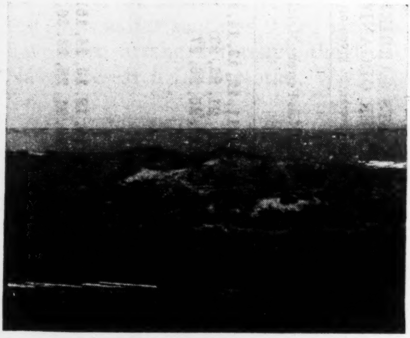

FIG. 2.—Test Site at Point Reyes, Calif., Looking West Toward the Pacific Ocean.

Newark, N. J.:

This severe industrial test site was established in 1956 to replace the Port Authority test site. The specimens are mounted at an angle of 30 deg from the horizontal and face the SSW at an azimuth of 193 deg (elevation, 11 ft).

Point Reyes, Calif.:

Point Reyes test site (see Fig. 2) was established in 1950 and is located 1930 ft from the Pacific Ocean behind low hills covered with salt grass and bushes. The specimens are mounted at an angle of 30 deg from the horizontal and face due west, toward the Pacific. The atmosphere here is characterized by salt spray and condensation exposure due to westerly winds and dense fogs which keep the specimens moist during most of the winter. In summer the area is very dry by day with frequent heavy fogs at night.

Kure Beach, N. C.:

The two exposure sites at Kure Beach (see Fig. 3) are under the direction of the International Nickel Co. and are located on the Cape Fear Peninsula 17 miles southeast of Wilmington, N. C. One test site is approximately 800 ft and the other approximately 80 ft from the Atlantic Ocean. The specimens are mounted 30 deg from the horizontal and face south at an azimuth of 177 deg at the 800-ft site. At the 80-ft site, the specimens parallel the beach at an azimuth of 110 deg. The 80-ft site is characterized by sea water spray falling directly on the test specimens. A discussion of the corrosive properties of these sites is to be found in the 1957 Edgar Marburg Lecture (**36**).

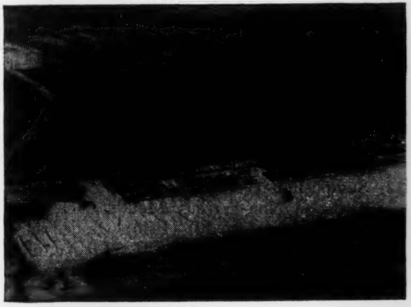

FIG. 3.—Test Sites at Kure Beach, N. C. (800 and 80 ft from the beach), Atlantic Ocean in the Foreground.

Freeport, Tex.:

In 1952 a test site was established on the Brazos River 3900 ft northwest of the Gulf of Mexico (see Fig. 4). The specimens are mounted at 30 deg from the horizontal and face southeast (azimuth of 144 deg). The climate in this area is noted for its consistently high humidity. The daytime relative humidity varies between 85 and 93 per cent in the summer and averages 80 per cent in the winter. The night-time humidity is almost 100 per cent all year with frequent heavy dews.

Tucson, Ariz.:

In 1931, a test site at Pheonix was established (for data on two Committee B-3 programs here, see Reference (**14**)); however, the test site had to be discontinued in 1950. In order to have available a dry desert atmosphere for ASTM exposure programs, another test site was established in 1952, two miles west of Tucson in an area with low shrubs on the Southwestern Forest and Range Experiment Station located on the eastern edge of the Coronado National Forest (elevation, 2560 ft). One acre is available for ASTM use. To date no construction has been made.

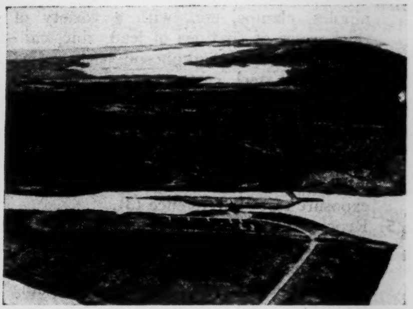

FIG. 4.—Test Site on the Intracoastal Waterway at the Brazos River near Freeport, Tex., Gulf of Mexico in the Background.

Black fence outlines test site area.

Respectfully submitted on behalf of the committee,

K. G. COMPTON,
Chairman.

S. F. ETRIS,
Secretary.

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(2) 1958 Annual Report of Committee A-5 on Corrosion of Iron and Steel. Latest exposure data on 1926 galvanized sheet tests at State College, Pa., *Ibid.*, Vol. 58, p. 116 (1958).

(3) Report of Committee A-5, Subcommittee VIII on Field Tests of Metallic Coatings, *Proceedings*, Am. Soc. Testing Mats., Vol. 29, Part I, p. 149 (1929). Program data for a series of hardware angles, tubes, nipples, clamps, etc., with a variety of thicknesses of coatings of lead, zinc, cadmium, aluminum, and phosphates on rolled and forged steel, cast, and malleable iron placed on exposure in 1929.

(4) Report of Committee A-5, Subcommittee XVI on Field Tests of Atmospheric Corrosion of Hardware, *Ibid.*, Vol. 57, p. 130 (1957). Latest data on the 1929 hardware exposure test of Reference (3).

(5) Report of Wire Inspection Committee on Field Tests of Wire and Wire Products, *Ibid.*, Vol. 39, p. 156 (1939). Program data on farm field fencing products including zinc, lead, and copper coated wire, and non-coated copper-bearing steel, and corrosion resistant steel wire placed on exposure in 1937.

(6) Report of Committee A-5, Subcommittee XV on Atmospheric Exposure Tests of Wire and Wire Products, *Ibid.*, Vol. 57, p. 99 (1957). Latest exposure data on the 1937 wire and field fence exposure test.

(7) Report of Committee A-5, Subcommittee VIII on Field Tests of Metallic Coatings, *Ibid.*, Vol. 36, Part I, p. 107 (1936). Cadmium and zinc and zinc-cadmium plated coatings on SAE 1010 steel exposed in 1932, 1936, and 1938.

(8) W. Blum, P. W. C. Strausser, A. Brenner, "Corrosion-Protective Value of Electrodeposited Zinc and Cadmium Coatings on Steel," *Journal of Research*, National Bureau of Standards, Vol. 16, No. 2, p. 185 (1936). A report on the 1932 program of Committee A-5, Subcommittee VIII on Field Tests of Metallic Coatings (same test as Reference (7)).

(9) Progress Report of a Subgroup of Committee B-3, Subcommittee VII on Corrosiveness of Various Atmospheric Test Sites as Measured by Specimens of Steel and Zinc, *Proceedings*, Am. Soc. Testing Mats., Vol. 53, p. 194 (1953). Interim test data given, exposures are now completed and a final report is in preparation.

(10) Report of Committee B-3, Subcommittee VIII on Galvanic & Electrolytic Corrosion, *Ibid.*, Vol. 39, p. 247 (1939). Galvanic couples involving aluminum, copper, iron, lead, nickel, tin, and zinc with the outer faces of the metal disks painted to confine the corrosion to the edges of the metal-to-metal surfaces of the couples, final report data of exposure begun in 1931.

(11) Report of Committee B-3, Subcommittee VI on Atmospheric Corrosion (Atmospheric Corrosion Test Program), *Ibid.*, Vol. 32, Part I, p. 226 (1932). Program data for 11 non-ferrous metals and alloys. Specimens placed on exposure in 1931 to determine change in weight and loss of strength and ductility by means of tension tests. Also see Reference (15).

(12) Symposium on Atmospheric Exposure Tests on Non-Ferrous Metals, Am. Soc. Testing Mats., p. 101 (1946). (Issued as a separate publication *ASTM STP No. 67.*) Reprinting of photomicrographs from report given in Reference (11).

(13) *Ibid.*, pp. 2, 16, 29, 46, and 57. Papers on the zinc alloys, nickel alloys, copper alloys, lead and tin alloys, and aluminum alloys utilized for the 1931 exposure test given in Reference (11).

(14) Report of Committee B-3, Subcommittee VI on Atmospheric Corrosion, Symposium on Atmospheric Corrosion of Non-Ferrous Metals, Am. Soc. Testing Mats., p. 3 (1956). (Issued as a separate publication *ASTM STP No. 175*). Twenty-year results on 1931 exposure test given in Reference (11).

(15) *Ibid.*, pp. 21, 67, 126, 135, and 141. Individual papers reviewing 20-year test results of the 1931 program given in Reference (11).

(16) Report of Committee B-3, Subcommittee VIII on Galvanic and Electrolytic Corrosion, *Proceedings*, Am. Soc. Testing Mats., Vol. 48, p. 167 (1948). Program data and results of 5-year exposure of two stainless steels (Types 304 and 316) coupled to 9 different metals exposed in 1941 (final

report on 15-year exposure is in preparation).

(17) H. O. Teeple, "Atmospheric Galvanic Corrosion of Magnesium Coupled to Other Metals," Symposium on Atmospheric Corrosion of Non-Ferrous Metals, Am. Soc. Testing Mats., p. 89 (1956). (Issued as a separate publication *ASTM STP No. 175.*) Data on the first series of aluminum and magnesium disk-type couples placed on exposure in 1950 (part I of a 3 part program).

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(20) G. R. Gohn and Lucille E. Menges, "Atmospheric and Indoor Aging Studies on Some Aluminum and Zinc-base Die-Casting Alloys," *Proceedings,* Am. Soc. Testing Mats., Vol. 46, p. 1064, (1946) and Report of Committee B-6, Subcommittee V on Exposure and Corrosion Tests, *Ibid.,* p. 244. Final report of 12 aluminum and 10 zinc die casting alloys exposed in 1929 for 15 years to determine loss in tensile strength, elongation, and Charpy impact.

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(24) L. H. Adam, "Atmospheric Exposure of Light Metals," *Ibid.,* Vol. 53, p. 227 (1953). Data on wrought and cast, structural aluminum and magnesium alloys, and 4 clad-aluminum specimens exposed in 1953 to determine reduction of mechanical properties.

(25) L. H. Adam and Marie Dougherty, "Atmospheric Exposure of Aluminum and Magnesium Sand and Permanent Mold Castings," *Ibid.,* Vol. 54, p. 270 (1954). Data from 1 year of exposure.

(26) Data from 3 years of exposure of test in Reference (**25**), *Ibid.,* Vol. 58, p. 194 (1958).

(27) L. H. Adam, "Atmospheric Exposure of Wrought Aluminum and Magnesium Alloys," ASTM *Proceedings,* Vol. 55, p. 284 (1955). Data from 1 year of exposure.

(28) W. Blum, P. W. C. Strausser, A. Brenner, "Protective Value of Nickel and Chromium Plating on Steel," *Journal of Research,* National Bureau of Standards, Vol. 13, No. 3, p. 331 (1934). Eighty variations of nickel, copper, and chromium cover coatings with some zinc and cadmium as intermediate coatings on SAE 1010 steel exposed in 1932 to determine the value of such coatings.

(29) W. Blum and P. W. C. Strausser, "Outdoor Exposure Tests of Electroplated Nickel and Chromium Coatings on Steel and Non-Ferrous Metals," *Ibid.,* Vol. 24, No. 4, p. 443 (1940). Over 135 variations of specimens to determine the value of coatings utilizing copper, nickel, and chromium on steel, copper, brass, iron, zinc, and zinc-base die-castings exposed in 1936.

(30) Report of Committee B-8, Subcommittee II on Performance Tests, "Atmospheric Exposure of Electroplated Lead and Zinc Coatings on Steel," *Proceedings,* Am. Soc. Testing Mats., Vol. 49, p. 220 (1949), and Vol. 55, p. 338 (1955). Results of exposure tests of electroplated coatings including various thicknesses of lead applied to steel, copper-plated steel, and copper; lead-tin alloy coatings; electroplated zinc; and hot-dipped lead, terne, and zinc exposed in 1944.

(31) A. H. DuRose, "Atmospheric Exposure of Electroplated Lead Coatings on Steel," Symposium on Properties, Tests, and Performance of Electrodeposited Metallic Coatings, Am. Soc. Testing Mats., p. 97 (1956). (Issued as a separate publication *ASTM STP No. 197.*) More detailed discussion of program in Reference (**30**).

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(33) C. H. Sample, "The Corrosion Behavior and Protective Value of Copper-Nickel-Chromium and Nickel-Chromium Coatings on Steel," Symposium on Properties, Tests, and Performance of Electrodeposited Metallic Coatings, Am. Soc. Testing Mats., p. 7 (1956). (Issued as a separate publication *ASTM STP No. 197*.) More detailed discussion, with photographs, of program in Reference (**32**).

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(37) Report of Committee B-6 on Die-Cast Metals, Exposure Tests of SC84A Aluminum Bars with Varying Zinc Content, Progress Report of Task Group of Subcommittee V on Exposure and Corrosion Tests, *Proceedings*, Am. Soc. Testing Mats. Vol. 57, p. 174 (1957). Three-year exposure report on a series of specimens of SC84A aluminum alloys with varying zinc content begun in 1953 for a period of 12 years.

(38) "Summary Report of Investigations of Aluminum-Base Die-Casting Alloys Containing 9.5 Per Cent Silicon and 0.5 Per Cent Magnesium or Containing 8 Per Cent Magnesium," *Ibid.*, Vol. 48, p. 191 (1948). Chemical composition and mechanical properties of alloys G2, SG2, and SG3, with results of salt spray tests and 2-yr atmospheric exposure data.

(39) Report of Committee B-6 on Die-Cast Metals and Alloys, *Ibid.*, Vol. 58, p. 167 (1958). Final 5 year exposure report of aluminum alloys (redesignated G8A and SG100A), placed on exposure in 1944 to determine loss in mechanical properties. See also Reference (**38**).

(40) C. H. Sample, A. Mendizza, and R. B. Teel, "A Comparison of the Corrosion Behavior and Protective Value of Electrodeposited Zinc and Cadmium Coatings on Steel," Symposium on Properties, Tests, and Performance of Electrodeposited Metallic Coatings, Am. Soc. Testing Mats., p. 49 (1956). (Issued as a separate publication *ASTM STP No. 197*.)

(41) A. L. Alexander, "Evaluation of Phosphate Coating over Electrodeposited Zinc," *Ibid.*, p. 66.

REPORT OF THE JOINT ASTM-ASME COMMITTEE

ON

EFFECT OF TEMPERATURE ON THE PROPERTIES OF METALS*

Meetings:

Meetings of the ASTM-ASME Joint Committee on the Effect of Temperature on the Properties of Metals, its panels and subcommittees were held during the past year in connection with the Annual Meetings of the ASTM in Atlantic City, N. J., during the week of June 16, 1957, and of The American Society of Mechanical Engineers in New York City the week of December 1, 1957.

Technical Sessions and Papers:

Two papers were presented at the ASTM Annual Meeting during the week of June 16, 1957:

"Embrittling Tendencies of Austenitic Superheater Materials at Elevated Temperatures," by J. H. Hoke, F. Eberle, and R. D. Wylie, Babcock & Wilcox Co.

"Thermal Shock Resistance of High-Temperature Alloys," by F. L. Muscatell, E. E. Reynolds, W. W. Dyrkacz, and J. H. Dalheim, Allegheny Ludlum Steel Corp.

A paper by W. B. Hoyt, "Strength of Welded Joints in Carbon Steel at Elevated Temperatures," reporting work sponsored by the Chemical and Petroleum Panel, was presented at a meeting of the ASME Petroleum Division at Tulsa, Okla., in September, 1957.

The following papers were presented at the Annual Meeting of the ASME during the week of December 1, 1957:

"Oxidation of Superheater Materials by High-Temperature Steam," by J. H. Hoke and F. Eberle, Babcock & Wilcox Co.

"Investigation of the Suitability of 18-8 (Type 304) Alloy for Superheater Service with Respect to Corrosion and Stress-Corrosion Behavior in Chloride-Bearing Steam Condensate," by F. Eberle, Thomas A. McNary, and Fred B. Snyder, Babcock & Wilcox Co.

"Properties of Cast Iron at Elevated Temperatures," by J. R. Kattus, Southern Research Institute.

The following papers sponsored by the committee are being presented at the ASTM Annual Meeting, June, 1958:

"The Creep Properties of Three Low-Shrinkage, Copper-Base Casting Alloys," by W. F. Simmons and J. G. Kura of Battelle Memorial Inst.

"Mechanical and Physical Properties of Three Low-Shrinkage, Copper-Base Casting Alloys," by J. G. Kura and R. M. Lang of Battelle Memorial Inst.

"Properties of Cast Iron at Elevated Temperatures," by J. R. Kattus and Bryan McPherson of Southern Research Inst.[1]

Membership:

Dr. Leo Schapiro, Douglas Aircraft Co., Inc., Chairman of the Aviation Panel, resigned during the year.

Finances:

The status of funds in the Committee's account as of April 1, 1958 is given in Table I.

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* Sixty-first Annual Meeting of the Society, June 22–27, 1958.

[1] The Kattus and McPherson paper is to be issued as a separate publication *ASTM STP No. 248*.

TABLE I.—STATUS OF FUNDS IN THE ACCOUNT OF THE ASTM-ASME JOINT COMMITTEE ON EFFECT OF TEMPERATURE ON THE PROPERTIES OF METALS AS OF APRIL 1, 1958.

Disbursements by Projects	*Appropriation*	*Total spent 3/31/58*	*Balance*	
Cash on hand, April 1, 1958				$19 728.65
Data and Publications Panel				
DP-6, Strength of Weldments	$ 3 136.00	$ 2 550.13	$ 585.87	
DP-8, Medium-Carbon, High-Chromium, and Plain-Carbon Steels	4 000.00 173.75	4 000.00	... 173.75	
DP-9, Aluminum and Magnesium Alloys	5 200.00	...	5 200.00	
DP-10, Physical Properties of Low and High Temperature	200.00	122.86	77.14	
DP-11 & 13, Continuing Data Survey and Punch Card Tabulation of Data	3 000.00	1 250.61	1 749.39	
Steam Power Panel				
SP-2, Elevated Temperature Properties of Cast Iron	20 000.00	20 000.09	closed	
SP-5, Use of Type 347 Stainless Steel in Main Steam Piping	5 000.00	...	5 000.00	
General Research Panel				
Creep Damage (University of Michigan)	2 000.00	...	2 000.00	
Cyclic Loading and Heating (Syracuse University)	2 000.00	1 999.76	closed	
Notch Sensitivity (Syracuse University)	3 000.00	2 787.89	closed	
Reproduction of Reports (Syracuse University)	400.00	400.00	closed	
Test Methods Panel				
Calibration Material	1 500.00	...	1 500.00	
			16 286.15	
Obligated and not spent, March 31, 1958				16 286.15
Estimated Unallocated Balance, March 31, 1958				3 442.50

NEW TENTATIVE

The committee recommends for publication as tentative the Recommended Practice for Conducting Creep and Time-for-Rupture Tension Tests of Materials, as appended hereto.[2]

REVISION OF STANDARD AND REVERSION TO TENTATIVE

The committee recommended revisions as indicated of the following recommended practices and their reversion to tentative:

Recommended Practice for Short-Time Elevated Temperature Tension Tests of Materials (E 21 – 43):[3] Revise as appended hereto.[2]

WITHDRAWAL OF TENTATIVE AND STANDARD

The committee recommends the withdrawal of the following tentative and standard since they are being replaced by the new tentative recommended practice referred to above:

Recommended Practice for:

Conducting Long-Time High-Temperature Tension Tests of Metallic Materials (**E 22 – 41**),[3] and

Conducting-Time for Rupture-Tension Tests of Metallic Materials (**E 85 – 50 T**).[3]

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[4]

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[2] The new and revised tentatives appear in the 1958 Book of ASTM Standards, Part 3.

[3] 1955 Book of ASTM Standards, Part 1.

[4] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

ACTIVITIES OF PANELS AND PROJECT SUBCOMMITTEES

Aviation Panel (*Leo Schapiro, chairman*):

The Task Group on Metal Honeycomb Panels completed a survey of existing test methods for testing honeycomb panels. The conclusion was that the present methods sponsored by ASTM Committee C-19 on Structural Sandwich Construction were not adequate for the aviation industry needs. The report recommended that either ASTM Committee C-19 or the Aircraft Industries Assn. provide the required test procedures.

The Task Group on Non-Tension Creep Testing is studying the desirability of providing a written test procedure for compression creep testing, for shear creep testing, and for bearing creep testing.

Data and Publications Panel (*George V. Smith, chairman*):

Project DP-6, Strength of Weldments.—This project has been completed and the data are being published by ASTM as a Special Technical Publication on "Elevated Temperature Properties of Weld-Deposited Metal and Weldments," *ASTM STP No. 226.*

Project DP-8, Medium-Carbon, High-Carbon, and Plain Carbon Steels.—The third and final phase of this survey, dealing with high-chromium steels, has been completed and is being published by ASTM as a Special Technical Publication, *ASTM STP No. 228.*

Project DP-9, Aluminum and Magnesium Alloys—A large number of data have now been received in this survey. The task group is undertaking to limit the data, which will be included in a final report, to those materials of commercial interest so that the resulting report will not be unduly large.

Project DP-12, Survey of Test Facilities.—The results of this survey have been published by the Titanium Metallurgical Laboratory of Battelle Memorial Institute.

Work is continuing on Project DP-10, Survey of Physical Properties of Low and High Temperatures; Project DP-11, Current and Continuing Data Survey; and Project DP-13, Panel Card Tabulation of Data.

Gas Turbine Panel (*C. T. Evans, Jr., chairman*):

Work is continuing on Project GTP-1, Fatigue; GTP-2, Corrosion Erosion; GTP-5, Thermal Shock; and GTP-10, Long-Time Creep and Rupture Testing. Several reports have been issued on these projects including the paper titled: "Thermal Shock Resistance of High-Temperature Alloys," which was presented at the ASTM Annual Meeting in June, 1957.

General Research Panel (*J. D. Lubahn, chairman*):

Literature surveys covering three subjects have been completed. The work was done under contract as follows:

(*1*) Creep Damage, $2000, with the University of Michigan,
(*2*) Notch Rupture Behavior, $2800, with Syracuse University, and
(*3*) Effect of Cyclic Loading, $2000, with Syracuse University.

Plans for publication of these surveys by the ASTM are under way.

A preliminary program was presented to determine methods of utilizing notch-rupture data to predict the life of notched machine parts at elevated temperature. The money value of the proposed investigation was estimated as $25,000. Contributions of material and testing facilities would substantially reduce the amount of money required.

Low Temperature Panel (*A. L. Tarr, chairman*):

Work on Project LT-2, "Literature Survey on the Mechanical Properties of

Useful Engineering Metals and Alloys at Low Temperatures," is being expedited. The task group handling Project LT-3, on a study of correlation of Charpy V-notch and keyhole data on materials used in pressure vessels and piping service, issued a final report on its work. The report stated that sufficient data were not available for a general correlation of Charpy V-notch and keyhole data for materials other than ship steels. This report completes the work of the task group, which was therefore discharged. The task group handling Project LT-4 on the use of cast iron at low temperatures reported that Subcommittee 26 of ASTM Committee A-3 on Cast Iron planned to undertake a similar study. There being no need for duplication of this work, the task group was discharged.

Chemical and Petroleum Panel (*C. L. Clark, chairman*):

Experimental work is continuing on an investigation covering weldments of low-chromium–molybdenum steels. A report on "Strength of Welded Joints in Carbon Steel at Elevated Temperatures" was completed and presented before the Petroleum Division of ASME at a meeting in Tulsa, Okla., September, 1957.

Steam Power Panel (*P. Brister, chairman*):

Work has been completed on Project SP-2, Elevated Temperature Properties of Cast Iron. The work was done under contract at the Southern Research Inst., Birmingham, Ala. A final report, an outline of which is appended hereto, will be presented at the ASTM Annual Meeting, June, 1958. Project SP-4, Elevated Temperature Properties of A 212-B Plate, is nearly completed and a final report will be issued for approval by the panel. A report on Project SP-5, Use of Type 347 Stainless Steel in Main Steam Piping, will be submitted to the panel for approval. This work is being privately financed by the International Nickel Co., General Electric Co., Westinghouse Electric Corp., and other investigators.

Considerable progress has been made on Project SP-6, Experience with Type 321 and 347 Stainless Steel in Superheater Tubes. Questionnaires were sent to all utilities having stainless steel in their superheaters, and the results are being analyzed. Work is under way on Project SP-7, Material Evaluation, the purpose of which is to evaluate materials as to their over-all suitability for use in steam power service.

Test Method Panel (*J. J. Kanter, chairman*):

The project for providing calibration specimens for creep-rupture testing is continuing. Tests are presently being made to determine whether the proposed material for the specimens meets all requirements. The panel submitted revisions to the Recommended Practices E 21, E 22, and E 85 as appended to this report.

This report has been submitted to letter ballot of the committee, which consists of 28 members; 15 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

V. T. MALCOLM,
Chairman.

HOWARD C. CROSS,
Secretary.

REPORT OF COMMITTEE C-1

ON

CEMENT*

Committee C-1 on Cement held two meetings during the year: in Atlantic City, N. J., on June 21, 1957, and in Fortin de las Flores, Veracruz, Mexico, on December 3, 1957. During the Mexico meetings the committee members had an opportunity to visit the cement mills of Cementos Veracruz, S. A.

The committee records with sorrow the deaths of two Honorary Members and two Active Members: L. W. Walter, an Honorary Member, joined Committee C-1 in 1914 and served as its Secretary from 1936 to 1942; H. S. Mattimore, also an Honorary Member, first joined the committee in 1926; H. L. Kennedy, a member since 1940, represented the Dewey & Almy Chemical Co.; and L. C. Smull, a member since 1951, represented the Riverside Cement Co.

In recognition of his long association with Committee C-1 and his outstanding contributions to its work, Frank H. Jackson was made an Honorary Member of the committee. Mr. Jackson, a member for over 35 years, had served the committee as secretary from 1926 to 1931, and as chairman from 1945 to 1950.

The officers elected for the ensuing term of two years are as follows:

Chairman, R. R. Litehiser.
Vice-Chairman, W. S. Weaver.
Secretary, W. J. McCoy.

Transfer of Jurisdiction.—In December 1957, Committees C-1 and C-9 agreed that responsibility for Tentative Methods of Test for Potential Alkali Reactivity of Cement-Aggregate Combinations (C 227 – 52 T) should be transferred from Committee C-1 to Committee C-9.

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1957 Annual Meeting, Committee C-1 presented to the Society, through the Administrative Committee on Standards, the following recommendations:

Revision of Tentative Method of Test for:

Calcium Sulfate in Hydrated Portland Cement Mortar (**C 265 – 56 T**),
Flexural Strength of Hydraulic Cement Mortars (**C 348 – 54 T**), and
Compressive Strength of Hydraulic Cement Mortars (Using Portions of Prisms Broken in Flexure (**C 349 – 54 T**).

These recommendations were accepted by the Standards Committee on December 13, 1957, and the revised tentative methods appear in the 1957 Supplement to the Book of ASTM Standards, Part 3.

REVISION OF TENTATIVES

The committee recommends revisions as indicated of the following tentative specifications and methods and their continuation as tentative:

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* Sixty-first Annual Meeting of the Society, June 22–27, 1958.

Standard Laboratory Condition.—For the purpose of promoting uniformity in laboratory atmospheres, the committee recommends that, where applicable, the following sentence be added to Section 3 of the various cement test methods: "The relative humidity of the laboratory shall be not less than 50 per cent."[1]

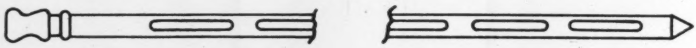

FIG. 1.—Tube Sampler for Bulk Cement.

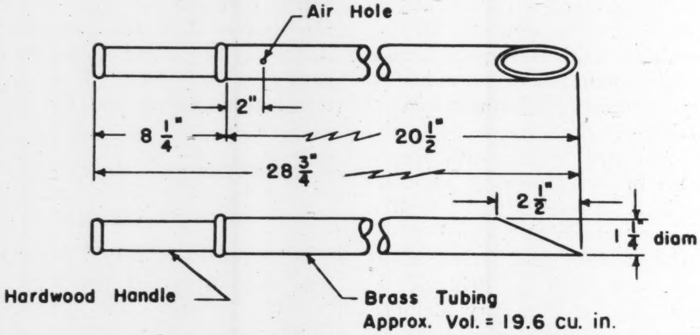

FIG. 2.—Tube Sampler for Packaged Cement.

Tentative Methods of Chemical Analysis of Portland Cement (C 114 – 57 T):[2]

New Section.—Add a new Section 30 to read as follows:

30. *Ignition Loss of Portland Blast-Furnace Slag Cement and Slag Cement.*—The methods given in Section 20 of C 114 – 53 are not suitable for determining the ignition loss of Portland-Blast Furnace Slag Cement and Slag Cement. This is normally true because the sulfide sulfur usually present in such cements will be oxidized to the sulfate in some degree and thus give an increase in sample weight. Therefore, on such samples use the following procedure: Determine the SO_3 by the method given in Section 16 of C 114 – 53 on a portion of the original sample. Determine the loss on ignition as described in Section 20 of C 114 – 53, using a single 15 min ignition period without checking for constant weight. After weighing, transfer the ignited material to a beaker, breaking it up with the flattened end of a glass rod. Determine the SO_3 by the method given in Section 16 of C 114 – 53. (Some of the acid is heated in the crucible to dissolve adhering material.) Correct the percentage loss on ignition by adding an amount equal to 0.8 times the increase in the percentage of SO_3 that developed during ignition.

Tentative Methods of Sampling Hydraulic Cement (C 183 – 55 T):[3]

Section 3.—Change the first sentence to read, "The cement may be sampled by any of the methods described in the following Paragraphs (*a*) to (*f*)."

Change Paragraphs (*c*) and (*d*) to read as follows:

(*c*) *From Bulk Storage and Bulk Shipments by Means of a Tube Sampler or Sampling Pipe.*—When the depth of the cement to be sampled does not exceed 7 ft, samples may be obtained by a tube sampler similar to that shown in Fig. 1. It is between 5 and 6 ft long and about 1$\frac{3}{8}$ in. in outside diameter and consists of two polished brass telescopic tubes with registering slots which are opened or closed by rotation of the inner tube, the outer tube being provided with a sharp point to facilitate penetration. Where applicable, for depths of cement greater than 7 ft, a sampling pipe activated by an air jet which is capable of removing samples from different depths in the cement may be used. Samples obtained with the tube sampler or sampling pipe shall be taken from well-distributed points and various depths of the cement so that the samples taken will represent the cement involved.

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[1] The tentatives involved are: C 185, C 227, C 243, C 265, C 266, and C 305.

[2] 1957 Supplement to Book of ASTM Standards, Part 3.

[3] 1955 Book of ASTM Standards, Part 3.

(*d*) *From Packaged Cement by Means of a Tube Sampler.*—The sampler shown in Fig. 2 should be inserted diagonally into the valve of the bag and the thumb placed over the air hole. The sampler should then be withdrawn. One sample should be taken from a bag in each 25 bbl or fraction thereof and combined to form the test sample.

Add two new Paragraphs (*e*) and (*f*) to read as follows:

(*e*) In all other cases, samples shall be taken from each 15 bbl or portion thereof in the lot and combined to form test samples. In the case of samples from trucks where the cement is being trucked from one mill, it is permissible to combine the samples from several trucks to form a test sample representing not more than 500 bbl. Samples of bulk shipments should be taken from well distributed points so as to be representative of the cement.

(*f*) *Protection of Samples.*—Immediately as samples are taken they shall be placed into moisture-proof, airtight containers to avoid moisture absorption and aeration of the sample.

New Figures.—Add a new Fig. 1, "Tube Sampler for Bulk Cement" and a new Fig. 2, "Tube Sampler for Packaged Cement" as indicated in the accompanying Figs. 1 and 2.

The above revisions will increase the scope of the method by providing new information about sampling devices and procedures, and sample containers.

Tentative Specifications for Portland Blast-Furnace Slag Cement (C 205 – 57 T):[2]

Section 1.—Change to read as follows:

1. *Scope.*—These specifications cover two basic types of hydraulic cement, with two optional provisions.

Type IS.—Portland blast-furnace slag cement for use in general concrete construction.

Type IS-A.—Air-entraining portland blast-furnace slag cement for use in general concrete construction.

When moderate sulfate resistance is desired, the suffix (MS) shall be added to the selected type designation. When moderate heat of hydration is desired, the suffix (MH) shall be added to the selected type designation. When both moderate sulfate resistance and moderate heat of hydration are desired, the suffix (MH) (MS) shall be added to the selected type designation.

Section 2.—In the second sentence of Paragraph (*b*), change "Section 3(*c*)" to read "Section 4(*c*)" and add the following new Note at the end of the section:

NOTE.—When the MS type (moderate sulfate resistance) is specified, the tricalcium aluminate content of the portland cement clinker constituent shall not exceed 8.0 per cent.

New Section.—Add a new Section 3 to read as follows, renumbering subsequent sections accordingly:

3. *Basis of Purchase.*—The purchaser should specify the type or types desired. When no type is specified, the requirements of Type IS shall govern.

Section 3.—Renumber as Section 4, and in the second sentence of the Note change "Section 3" to read "Section 4."

In Paragraph (a), change "Section 4" to read "Section 5."

Section 4.—Renumber as Section 5 and in Paragraph (a) change the first sentence to read "Portland blast-furnace slag cement of the type specified shall conform to the chemical requirements prescribed in Table I."

Section 5.—Renumber as Section 6 and change to read as follows:

6. *Physical Requirements.*—Portland blast-furnace slag cement of the type specified shall conform to the applicable physical requirements prescribed in Table II. When the type of cement is not specified, the requirements of Type IS shall govern.

Table II.—Change to read as indicated in the accompanying Table I.[4]

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[4] The table appears in the revised specifications; see 1958 Book of ASTM Standards, Part 4.

Section 11.—Renumber as Section 12 and add the following new Paragraph (*f*), relettering the subsequent paragraphs accordingly:

(*f*) *Time of Setting by Gillmore Needles.*—Method of Test for Time of Setting of Hydraulic Cement by Gillmore Needles (ASTM Designation: C 266).

Reletter the present Paragraph (*f*) as (*g*) and change the heading to read "Time of Setting by Vicat Needle."

Add a new paragraph (*k*) to read as follows:

(*k*) *Heat of Hydration.*—Method of Test for Heat of Hydration for Portland Cement (ASTM Designation: C 186).

The principal purpose of the revisions is to increase from two to eight the number of types of this cement that are available to the purchaser.

Tentative Specifications for Air-Entraining Additions for Use in the Manufacture of Air-Entraining Portland Cement (C 226 – 54 T):[3]

Section 3(c).—Change the last sentence of the second paragraph to read "The air contents of the concrete containing the reference addition and the concrete containing the proposed addition shall agree within 0.5 per cent."

Add the following to this section:

The reference addition, used as an admixture, is intended for use with blank cements only in concrete for freezing-and-thawing tests to establish the durability factor by means of which the concretes containing the addition under test may be evaluated.

Preparation of Standard Reference Solutions: Place 50 g. (total solids in the case of solution or pastes) of the designated reference addition in 500 ml. of freshly distilled water in a 1.000-liter flask and mix thoroughly until the solids are completely dissolved or the paste or solution is uniformly diluted. After surface foam has been dissipated, dilute to 1.000 liter and mix thoroughly.

In the case of Vinsol resin, the neutralized solution shall be made as follows: Dissolve 7.50 g. cp sodium hydroxide in 100 ml. distilled water. Add a few drops of this solution to 300 to 350 ml. of distilled water contained in a 600-ml. beaker. Add 50.00 g. dry, unneutralized Vinsol resin in pulverized form to the beaker and stir until all of the resin is wetted and well dispersed. Then add all of the sodium hydroxide solution to this suspension and stir until all of the resin is in solution. Transfer to a measuring flask, dilute to 1.000 liter and mix thoroughly. From this stock standard solution prepare a dilute standard solution by diluting 100 ml. of the stock solution to 1.000 liter.

Section 4(a).—Add a new paragraph to read as follows:

The quantity of sample shall be not less than 8 standard sacks for cement containing the proposed addition and not less than 10 sacks of the corresponding blank cement. If the testing agency wishes to make direct comparisons between the proposed and reference additions as to compressive and flexural strength, bond to steel, or bleeding and volume change, then the quantity of blank cement shall be increased to 16 sacks.

Section 9(a).—Change the first sentence to read "Six test specimens from each condition of concrete to be compared shall be made for each test and age."

Section 10(b).—Add a new second sentence to read "Flexural strength specimens shall be tested at the ages of 3, 7, and 28 days, 3 months, and 1 year, except that for Type III cement a test shall be made also at 1 day."

Tentative Specifications for Portland-Pozzolan Cement (C 340 – 57 T):[2]

Section 13(l).—Change Paragraph (*2*) to read as follows:

(*2*) *Proportioning and Mixing.*—The mortar shall contain two parts of the oven dry pozzolan, one part of hydrated lime, and nine parts of graded standard sand by weight. The amount of mixing water, measured in milliliters, shall be such as to produce a flow of 110 ± 5 per cent flow in accordance with the procedure described in Section 9 of Method C 109.

A sufficient quantity of mortar shall be made to mold at least three cylindrical specimens 2 in. in diameter and 4 in. in height. The dry pozzolan and lime shall be put together and shaken vigorously in a covered container for 2 min. The mortar shall be mixed in accordance with Section 5 of Method C 305, except that Section 5 (2) shall be changed to read: "Add the pozzolan-lime mixture to the water; then start the mixer and mix at the slow speed (140 ± 5 rpm) for 30 sec."

Change the first sentence of Paragraph (*3*) to read, "The molds shall be cylindrical and shall have an inside diameter of 2 ± $\frac{1}{8}$ in. and a height of 4 ± $\frac{1}{8}$ in."

ADOPTION OF TENTATIVE AS STANDARD WITH REVISION

The committee recommends that the Tentative Specifications for Slag Cement (**C 358 – 57 T**)[2] be approved for reference to letter ballot of the Society for adoption as standard with the following revision:

Section 13(*b*).—Delete the words, "except that the sample shall be fused with sodium carbonate to insure solution, except in the determination of additions."

REVISION OF STANDARDS, IMMEDIATE ADOPTION

The committee recommends revisions for immediate adoption in the following standards, and accordingly asks for the necessary nine-tenths affirmative vote at the Annual Meeting in order that these revisions may be referred to letter ballot of the Society.

Standard Laboratory Condition.—For the purpose of promoting uniformity in laboratory atmospheres, the committee recommends that, where applicable, the following sentence be added to Section 3 of the various cement test methods: "The relative humidity of the laboratory shall be not less than 50 per cent."[5]

Standard Specifications for Masonry Cement (C 91 – 57):[3]

In order to improve the procedure for making air content determinations, the following revisions are recommended:

Section 24.—Add a new Paragraph (*d*) as follows, relettering the present paragraph (*d*) as (*e*):

(*d*) *Tapping Stick.*—A tapping stick made of maple wood and having a square cross-section of $\frac{5}{8}$ in. and a length of 6 in.

Section 25(*b*).—Change the sixth sentence to read "After the measure has been filled and spaded in the above prescribed manner, tap the sides of the measure lightly with the flat side of the tapping stick once each at five different points at approximately equal spacing around the outside of the measure in order to preclude entrapment of extraneous air."

Standard Method of Test for Compressive Strength of Hydraulic Cement Mortars (Using 2-in. Cube Specimens) (C 109 – 56):[3]

Section 13.—In order to promote uniformity in the reporting of test results, add the sentence "The Compressive strength of all acceptable test specimens (see Section 14) made from the same sample and tested at the same period shall be averaged and reported to the nearest 10 psi."

Standard Methods of Chemical Analysis of Portland Cement (C 114 – 53):[2]

Section 28(*a*).—Change to read as follows, retaining the present Notes 1 and 2:

28. (*a*) To 1 g. of the sample (Note 1), add 25 ml. of cold water. Disperse the cement in the water and while swirling the mixture, quickly add 5 ml. of HCl. If necessary, warm the solution gently and grind the material with the flattened end of a glass rod for a few minutes until it is evident that decomposition of the cement is complete (Note 2). Dilute the solution to 50 ml. with hot water (near boiling) and heat the covered mixture rapidly to near boiling by means of a high-temperature hot plate. Then digest the covered mixture for 15 min. at a temperature just below boiling (Note 3). Filter the solution into a 400-ml. beaker,

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[5] The standards involved are: C 109, C 151, C 187, C 190, and C 191.

wash the beaker, paper and residue thoroughly with hot water, and reserve the filtrate for the sulfur trioxide determination, if desired (Note 4). Transfer the filter paper and contents to the original beaker, add 100 ml. of hot (near boiling) NaOH solution (10 g. per liter) and digest at a temperature just below boiling for 15 min. During the digestion, occasionally stir the mixture and attempt to macerate the filter paper. Acidify the solution with HCl using methyl red as the indicator and add an excess of 4 or 5 drops of HCl. Filter and wash the residue at least 14 times with hot NH_4NO_3 solution (20 g. per liter) making certain to wash the entire filter paper and contents during each washing. Ignite the residue in a tared crucible at 900 to 1000 C., cool in a desiccator, and weigh.

Add the following new Notes 3 and 4:

NOTE 3.—In order to keep the solutions closer to the boiling temperature, it is recommended that these digestions be carried out on an electric hot plate rather than on a steam bath.

NOTE 4.—Continue with the sulfur trioxide determination (Section 16 or 44) by diluting to 250 or 200 ml. as required by the appropriate section.

Section 28(c).—Change to read as follows:

(c) *Calculations.*—Calculate the percentage of the insoluble residue to the nearest 0.01 by multiplying the weight of the residue (corrected for the blank) in grams by 100.

Footnote 9.—In the last sentence, change "NH_4Cl" to read "NH_4NO_3."

These revisions are proposed in order to improve the reproducibility of results. No basic change in method is involved.

Standard Method of Test for Fineness of Portland Cement by the Turbidimeter (C 115 - 53):[3]

In order to effect some helpful changes in requirements for apparatus and to clarify and refine certain test techniques, the following revisions are recommended:

Section 3.—Change Paragraph (*a*) to read as follows:

(*a*) *Source of Light.*—The source of light (Fig. 1) shall consist of a 3 to 6 candlepower, concentrated filament electric lamp operated by a source of constant emf. The lamp shall be mounted rigidly in the socket. A clean, bright parabolic reflector shall be rigidly mounted behind the lamp, focused so that approximately parallel rays of light will pass through the sedimentation tank and impinge upon the photoelectric cell. The light intensity shall be regulated by two rheostats of approximately 6 and 30 ohms, respectively, and shall possess such characteristics that uniform changes in light intensity may be obtained over the full range of resistance. They shall be mounted in series with the lamp but in parallel with each other.

In the third sentence of Paragraph (*d*), change "±0.1 in. in height" to read "±0.15 in. in height."

In the first sentence of Paragraph (*g*), after "raised or lowered" add the words "by two connected lead screws."

Section 4.—Change to read as follows:

4. The microammeter shall have a range of 0 to 50 microamperes, shall be readable to 0.1 microampere and accurate to ±0.5 per cent of full scale value at any part of the scale at 25 C. The internal resistance of the microammeter shall be between 50 and 150 ohms. The microammeter shall not be mounted upon a working surface containing or consisting of iron or steel or near other magnetic influence.

Section 8.—Add the following to the end of the Section:

The graduation lines on the buret shall be complete circles. A filter made of No. 325 wire cloth shall be used with the timing buret and a cover shall be placed over the top of the buret when it is not in use.

Section 9.—Add the sentence "The kerosine shall not be reused."

Section 10(b).—Change to read as follows, retaining the present Notes:

(*b*) *Calibration of No. 325 Sieve.*—Connect the spray nozzle and pressure gage to a water supply line and adjust the pressure on the spray nozzle to 10 ± 1 psi. Place 1 g. of the current lot of National Bureau of Standards standard sample No. 114^b on the No. 325 (44 micron) sieve. The sample shall be wetted by introducing the sieve into the spray at an angle so as to prevent loss of material. The sieve shall be held with the nozzle just within the upper rim, and moved so as to make a circuit about the nozzle about once per second. Wash for 1 min. After removing from the spray, rinse the sieve and contents carefully with distilled water and blot excess water from the bottom of the sieve by placing upon a damp cloth. Dry the sieve and residue in an oven or over a hot plate (Note 2) supporting the sieve so as to allow circulation of air from below, brush the residue from the sieve, and weigh on an analytical balance. The sieve correction factor is the difference between the amount of residue obtained in this manner and the amount of residue indicated by the fineness specified for the standard sample, expressed as a percentage of the former residue (see Note 3; also Table III, Footnote *a*).

Section 13(b).—In the second sentence, after the word "axis" add the phrase, "through the center of the tank."

Standard Methods of Test for Autoclave Expansion of Portland Cement (C 151 – 56):[3]

Section 4(c).—Change to read as follows:

(*c*) *Molding Specimens*.—Immediately following completion of mixing, the test specimen shall be molded in two approximately equal layers, each layer being compacted with the thumbs or forefingers by pressing the paste into the corners, around the reference inserts, and along the surface of the mold until a homogeneous specimen is obtained. After the top layer has been compacted, the paste shall be cut off flush with the top of the mold with a thin-edged trowel and the surface smoothed with a few strokes of the trowel. During the operations of mixing and molding, the hands shall be protected by rubber gloves.

This revision would eliminate any uncertainties about the technique to be used in molding test specimens.

REAPPROVAL OF STANDARDS

The committee recommends reapproval of the following two standards which have been published for six years or longer without revision:

Standard Method of Test for:

Fineness of Hydraulic Cement by the No. 200 Sieve (**C 184 – 44**), and
Specific Gravity of Hydraulic Cement (**C 188 – 44**).

TENTATIVES CONTINUED WITHOUT REVISION

The committee recommends that the following tentative methods be continued without revision. All are subjects of current study within the committee.

Tentative Method of Test for:

Air Content of Hydraulic Cement Mortar (**C 185 – 55 T**),
Time of Setting of Hydraulic Cement by Gillmore Needles (**C 266 – 51 T**),
Mechanical Mixing of Hydraulic Cement Mortars of Plastic Consistency (**C 305 – 55 T**), and
False Set of Portland Cement (**C 359 – 56 T**).

EDITORIAL CHANGES

The committee recommends editorial changes as indicated in the following standards and tentatives:

Method of Test for Compressive Strength of Hydraulic Cement Mortars (Using 2-in. Cube Specimens) (C 109 – 56):[3]

Section 10.—In the fifth sentence, change "Section 2(*i*)" to read "Section 2(*h*)."

Standard Methods of Test for Autoclave Expansion of Portland Cement (C 151 – 56):[3]

Section 6.—Change to read as follows:

6. The difference in length of the test specimen before and after autoclaving shall be expressed as per cent of the effective gage length and shall be reported to the nearest 0.01 per cent. The percentage of increase in length shall be reported as the autoclave expansion; a decrease in length shall be indicated by a minus sign prefixed to the per cent value.

Tentative Specifications for Air-Entraining Additions for Use in the Manufacture of Air-Entraining Portland Cement (C 226 – 54 T):[3]

Section 1.—Change to read as follows:

1. These specifications cover the requirements and methods of test for establishing the suitability of a material for use as an air-entraining addition to be interground with the clinker in

the manufacture of air-entraining portland cement under the Standard Specifications for Air-Entraining Portland Cement (ASTM Designation: C 175).

Tentative Specifications for Flow Table for Use in Tests of Hydraulic Cements (C 230 – 57 T):[3]

Section 4.—Change the fourth sentence to read "The table should be raised and permitted to drop a dozen or more times just prior to use if it has not been operated for some time."

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[6]

This report has been submitted to letter ballot of the committee, which consists of 90 voting members; 71 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

R. R. LITEHISER,
Chairman.

W. S. WEAVER,
Secretary.

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[6] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

REPORT OF COMMITTEE C-2
ON
MAGNESIUM OXYCHLORIDE AND MAGNESIUM OXYSULFATE CEMENTS*

Committee C-2 on Magnesium Oxychloride and Magnesium Oxysulfate Cements met once during the year: on June 18, 1957, in Atlantic City, N. J. during the Annual Meeting of the Society.

The committee consists of 17 members, of whom 7 are classified as producers, 5 as consumers, and 5 as general interest members.

The officers elected for the ensuing term of two years are as follows:

Chairman, E. S. Newman.
Vice-Chairman, J. B. James.
Secretary, K. M. Berg.

ADOPTION OF TENTATIVES AS STANDARD WITHOUT REVISION

The committee recommends that the following tentatives be approved for reference to letter ballot of the Society for adoption as standard without revision:

Tentative Definition of Terms Relating to:

Magnesium Oxychloride and Magnesium Oxysulfate Cements (**C 376 – 55 T**).[1]

Tentative Method of Test for:

Yield of Magnesium Oxychloride Cement (Field Test) (**C 388 – 56 T**),[2] and

Yield of Magnesium Oxychloride Cement (Laboratory Test) (**C 389 – 56 T**).[2]

REAPPROVAL OF STANDARDS

The committee recommends the reapproval of the following standards which have stood for six years without revision:

Standard Methods for:

Chemical Analysis of Magnesium Sulfate, Technical Grade (**C 244 – 52**),[1]
Chemical Analysis of Magnesium Chloride (**C 245 – 52**),[1]
Physical Testing of Magnesia for Magnesium Oxychloride Cements (**C 246 – 52**),[1] and
Mixing Magnesium Oxychloride Cement Compositions with Gaging Solution (for Preparation of Specimens for Laboratory Tests) (**C 251 – 52**).[1]

Standard Method of:

Slump Test for Field Consistency of Magnesium Oxychloride Cement (**C 249 – 52**).[1]

Standard Specifications and Method for:

Field Determination of Specific Gravity of Gaging Solutions for Magnesium Oxychloride Cements (**C 250 – 52**).[1]

Standard Method of Test for:

Bulk Density of Magnesium Oxychloride Cements (**C 248 – 52**),[1]
Linear Contraction of Magnesium Oxychloride Cements (**C 252 – 52**),[1]
Linear Change of Magnesium Oxychloride Cements (**C 253 – 52**),[1]
Setting Time of Magnesium Oxychloride Cements (**C 254 – 52**),[1]
Consistency of Magnesium Oxychloride Cements by the Flow Table (**C 255 – 52**),[1]
Flexural Strength of Magnesium Oxychloride Cements (Using Simple Bar with Two-Point or Single-Point Loading) (**C 256 – 52**),[1] and
Compressive Strength of Magnesium Oxychloride Cements (**C 257 – 52**).[1]

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* Sixty-first Annual Meeting of the Society, June, 22–27, 1958.

[1] 1955 Book of ASTM Standards, Part 3.

[2] 1956 Supplement to Book of ASTM Standards, Part 3.

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[3]

This report has been submitted to letter ballot of the committee, which consists of 17 members; 13 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

E. S. NEWMAN,
Chairman.

K. M. BERG,
Secretary.

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[3] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

REPORT OF COMMITTEE C-3

ON

CHEMICAL-RESISTANT MORTARS*

Committee C-3 on Chemical-Resistant Mortars held three meetings during the year: on June 19, 1957, in Atlantic City, N. J.; on October 31 and November 1, 1957, in Suffern, N. Y.; and on April 1, 1958, in Philadelphia, Pa.

The committee consists of 25 members, of whom 11 are classified as producers, 7 as consumers, 7 as general interest members, and 2 as consulting members.

The officers elected for the ensuing term of two years are as follows:

Chairman, J. R. Allen.

Vice-Chairman, W. H. Burton.

Secretary, E. A. Reineck.

Members-at-Large of Advisory Committee: Beaumont Thomas, F. H. Buckley, and W. A. Severance.

NEW TENTATIVES

The committee recommends for publication as tentative the following methods as appended hereto:[1]

Method of Test for:

Working and Initial Setting Times of Chemically-Setting, Silicate Type Chemical-Resistant Mortars, and

Absorption and Apparent Porosity of Chemical-Resistant Mortars.

TENTATIVE CONTINUED WITHOUT REVISION

The committee recommends the continuation without revision of Tentative Recommended Practice for Installing Sulfur Mortar Joints (**C 386 – 56 T**).

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[2]

SUBCOMMITTEE ACTIVITIES

Subcommittee S-1 on Silicate Mortars (R. F. Strigle, chairman) is attempting to establish limits on mechanical properties of silicate mortars, preparatory to drafting a specification.

Subcommittee S-4 on Hydraulic Mortars (W. C. Hansen, chairman) is cooperating with Subcommittee T-4 in revising the Tentative Recommended Practice for Use of Hydraulic Cement Mortars in Chemical-Resistant Masonry (C 398 – 57 T) as the result of comments from industry.

Subcommittee T-1 on Mechanical Properties (J. R. Allen, chairman) is working on a test method for determining flexural strength of silicate mortars.

Subcommittee T-2 on Physical Properties (R. S. Mercer, chairman) is studying the service temperature limits of chemical-resistant mortars as a background for selecting test methods to measure thermal expansion and shrinkage.

The subcommittee prepared the proposed Method of Test for Working and Initial Setting Times of Silicate-Type Chemical-Resistant Mortars, and the proposed Method of Test for Absorption

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*** Sixty-first Annual Meeting of the Society, June 22–27, 1958.**

[1] The new tentatives appear in the 1958 Book of ASTM Standards, Part 4.

[2] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

and Apparent Porosity of Chemical-Resistant Mortars, referred to earlier in this report.

Subcommittee T-3 on Chemical Properties (G. P. Gabriel, chairman) prepared a draft of a method of test for chemical-resistance of mortars. The test uses weight change and appearance of the exposed specimens and test solutions as guides for selection of mortars for particular applications. The draft will be expanded to include compressive strength as an added criteria.

Subcommittee T-4 on Recommended Practices and Identification (W. H. Burton, chairman) is revising the Tentative Recommended Practice for Use of Hydraulic Cement Mortars in Chemical-Resistant Masonry (C 398 - 57 T) as the result of comments from industry. The revision may also add recommendations on constructions other than floors.

The subcommittee plans to add a section on chemical-resistance to the recommended practices on resin, sulfur, and hydraulic mortars.

This report has been submitted to letter ballot of the committee, which consists of 25 members; 19 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

BEAUMONT THOMAS,
Chairman.

E. A. REINECK,
Secretary.

REPORT OF COMMITTEE C-4
ON
CLAY PIPE*

Committee C-4 on Clay Pipe held one meeting during the year: in Atlantic City, N. J., on June 18, 1957, during the Annual Meeting of the Society.

The officers elected for the ensuing term of two years are as follows:

Chairman, D. G. Miller.
Vice-Chairman, C. R. Velzy.
Secretary, R. G. Scott.

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1957 Annual Meeting, Committee C-4 presented to the Society through the Administrative Committee on Standards the following recommendations:

Revision of Tentative Specifications for:

Extra Strength Clay Pipe (**C 200 - 55 T**),
Extra Strength Ceramic Glazed Clay Pipe (**C 278 - 55 T**),

Revision and Reversion to Tentative of Standard Specifications for:

Standard Strength Ceramic Glazed Clay Sewer Pipe (**C 261 - 54**),
Standard Strength Clay Sewer Pipe (**C 13 - 54**), and
Standard Strength Perforated Clay Pipe (**C 211 - 50**).

These recommendations were accepted by the Standards Committee on September 13, 1957, and the revised Specifications appear in the 1957 Supplement to Book of ASTM Standards, Part 3.

AMERICAN STANDARDS

Three of the above specifications had been accorded status as American Standard by the American Standards Assn., namely, C 13, C 211, and C 261. The revised specifications were accordingly presented to the ASA and they were approved as American Standard on March 25, 1958.

This report has been submitted to letter ballot of the committee, which consists of 27 members; 24 members returned their ballots, of whom 23 have voted affirmatively and 1 negatively.

Respectfully submitted on behalf of the committee,

D. G. MILLER,
Vice-Chairman.

R. G. SCOTT,
Secretary.

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* Sixth-first Annual Meeting of the Society, June 22–27, 1958.

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee C-4 presented to the Society through the Administrative Committee on Standards the following recommendations:

New Tentative Specification for:

Jointing Vitrified Clay Pipe (C 425 – 58 T).

Revision and Reversion to Tentative of Recommended Practice for:

Installing Vitrified Clay Sewer Pipe (C 12 – 54).

These recommendations were accepted by the Standards Committee on September 9, 1958, and the new tentative specification and revised recommended practice appear in the 1958 Book of ASTM Standards, Part 5.

REPORT OF COMMITTEE C-7

ON

LIME*

Committee C-7 on Lime held two meetings during the year: on June 18, 1957 in Atlantic City, N. J., during the Annual Meeting of the Society, and on February 14, 1958, in St. Louis, Mo. during ASTM Committee Week.

The committee has adopted new By-Laws. Committee C-7 relinquished representation on Committee D-19 on Industrial Water. The committee also relinquished joint jurisdiction of the Tentative Specification for Inorganic Aggregates for Use in Gypsum Plaster (C 35 - 57 T), this standard now being under the sole jurisdiction of Committee C-11 on Gypsum.

The committee consists of 52 members, of whom 25 are classified as producers, 7 as consumers, 18 as general interest, and 2 honorary members.

The officers elected for the ensuing term of two years are as follows:

Chairman, J. A. Murray.

Vice-Chairmen, W. C. Voss, R. K. Thomas.

Secretary, L. E. Johnson.

NEW TENTATIVE

The committee recommends for publication as tentative the Proposed Specification for Quicklime and Hydrated Lime for Sand-Lime Products as appended hereto.[1]

ADOPTION OF TENTATIVE REVISION AS STANDARD

The committee recommends that the tentative revision of Section 18 of the Methods of Physical Testing of Quicklime and Hydrated Lime (**C 110 - 57**)[2] be approved for reference to letter ballot of the Society for adoption as standard.

REVISIONS OF STANDARD, IMMEDIATE ADOPTION

The committee recommends for immediate adoption revisions as follows of the Standard Methods of Chemical Analysis of Limestone, Quicklime, and Hydrated Lime (**C 25 - 47**),[2] and accordingly asks for the necessary nine-tenths affirmative vote at the Annual Meeting in order that the revisions may be referred to letter ballot of the Society:

Section 27.—Add a new Paragraph (*c*) to read as follows:

> (*c*) Combined water in quicklime is calculated from loss on ignition determination and carbon dioxide determination, as follows:
>
> $$\text{Loss on ignition} - CO_2 = \text{combined } H_2O$$
>
> Combined water in hydrated lime is calculated from loss on ignition determination, carbon dioxide determination, and mechanical moisture determination, as follows:
>
> $$\text{Loss on ignition} - (CO_2 + \text{mechanical moisture}) = \text{combined } H_2O$$

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* Sixty-first Annual Meeting of the Society, June 22–27, 1958.

[1] The new tentative appears in the 1958 Book of ASTM Standards, Part 4.

[2] 1955 Book of ASTM Standards, Part 3.

REAPPROVAL OF STANDARDS

The committee recommends the reapproval of the following standards which have stood for six or more years without revision:

Standard Specifications for:

Normal Finishing Hydrated Lime (**C 6 - 49**),
Special Finishing Hydrated Lime (**C 206 - 49**), and
Hydrated Lime for Masonry Purposes (**C 207 - 49**).

TENTATIVE CONTINUED WITHOUT REVISION

The committee recommends the continuation without revision of the Tentative Specification for Fly Ash for Use as a Pozzolanic Material with Lime (**C 379 - 56 T**).

EDITORIAL CHANGES

The committee recommends editorial changes as follows in the Standard Methods of Chemical Analysis of Limestone, Quicklime, and Hydrated Lime (**C 25 - 47**):[2]

Section 26(b).—In the first sentence change "13.7 ml of HCl" to read "15.7 ml of HCl."

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[3]

This report has been submitted to letter ballot of the committee, which consists of 52 members; 38 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

J. A. MURRAY,
Chairman.

L. E. JOHNSON,
Secretary.

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[3] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

REPORT OF COMMITTEE C-8
ON
REFRACTORIES*

Committee C-8 on Refractories held two meetings during the year: the ninety-second at Bedford Springs, Pa., on October 3, 1957; and the ninety-third at St. Louis, Mo., on February 14, 1958.

The committee consists of 46 members, of whom 24 are classified as producers, 16 as consumers, and 6 as general interest members.

The officers elected for the ensuing term of two years are as follows:

Chairman, J. J. Hazel.
Vice-Chairman, S. M. Phelps.
Secretary, L. J. Trostel.

NEW TENTATIVES

The committee recommends for publication as tentative the following method and classifications as appended hereto:[1]

Method of Test for:

Thermal Conductivity of Castable Refractories.

Classification of:

Silica Refractory Brick, and
Fireclay and High Alumina Refractory Brick. This constitutes a tentative revision of the Standard Classification of Fireclay Refractories (C 27 - 56).

TENTATIVE REVISIONS OF STANDARDS

The committee recommends tentative revisions as indicated of the following standards:

Definition of Terms Relating to Refractories (C 71 - 55):[2]

Revise the following three tentative revisions of definitions to read:

Raw Refractory Dolomite.—Natural dolomite which is sufficiently free of fluxing constituents and in proper form for use as a refractory material or in refractory products.

Calcined Refractory Dolomite.—Raw refractory dolomite which has been heated to a temperature sufficiently high to decompose the carbonate structure and remove chemically combined volatile constituents.

Dead-Burned Refractory Dolomite.—Raw refractory dolomite which has been heated to a temperature sufficiently high and for a long enough time to decompose the carbonate structure so as to form primarily calcia and periclase in a matrix that provides resistance to subsequent hydration and recombination with carbon dioxide.

Specification for Fireclay-Base Castable Refractories for Boiler Furnaces and Incinerators (C 213 - 55):[2]

Table I.—Change to read as indicated in the accompanying Table I.

ADOPTION OF TENTATIVE AS STANDARD WITHOUT REVISION

The committee recommends that the Tentative Method of Test for Bulk Density of Granular Refractory Materials (**C 357 - 55 T**)[2] be approved for reference to letter ballot of the Society for adoption as standard without revision.

ADOPTION OF TENTATIVE REVISIONS AS STANDARD

The committee recommends that the tentative revisions of the following standards be approved for reference to letter ballot of the Society for adoption as standard:

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* Sixty-first Annual Meeting of the Society, June 22-27, 1958.

[1] The new tentatives appear in the 1958 Book of ASTM Standards, Part 5.

[2] 1955 Book of ASTM Standards, Part 3.

Classification of Fireclay Refractories (C 27 - 56),[3]

Method for Basic Procedure in Panel Spalling Test for Refractory Brick (C 38 - 52)[2] (tentative revisions of Sections 5 and 6 only), and

Specification for Fireclay-Base Castable Refractories for Boiler Furnaces and Incinerators (C 213 - 55).[2]

WITHDRAWAL OF TENTATIVE REVISION

The committee recommends the withdrawal of the portion of the tentative revision, issued June, 1956,[3] of the Standard Definition of Terms Relating to Refractories (C 71 - 55),[2] consisting of definitions for abutment, acid proof brick, American bond, arch, arch brick, air ramming, and air set.

TABLE I.—REQUIREMENTS FOR FIRECLAY-BASE CASTABLE REFRACTORIES.

(Revision of Table I, Specification C 213)

Test Requirements	Class B	Class C
Permanent linear shrinkage, not more than 1.5 per cent after heating at	2300 F (1260 C)	2500 F (1370 C)
Modulus of rupture, not less than 300 psi after	drying at 220 to 230 F (105 to 110 C)	

REAPPROVAL OF STANDARDS

The committee recommends the reapproval of the following standards which have stood for six or more years without revision:

Standard Method of:

Testing Refractory Brick Under Load at High Temperatures (C 16 - 49),

Panel Spalling Test for High-Duty Fireclay Brick (C 107 - 52),

Panel Spalling Test for Super Duty Fireclay Brick (C 122 - 52), and

Panel Spalling Test for Fireclay Plastic Refractories (C 180 - 47).

Standard Methods of Test for:

Apparent Porosity, Water Absorption, Apparent Specific Gravity, and Bulk Density of Burned Refractory Brick (C 20 - 46),

Sieve Analysis and Water Content of Refractory Materials (C 92 - 46),

Reheat Change of Refractory Brick (C 113 - 46),

Size and Bulk Density of Refractory Brick (C 134 - 41),

True Specific Gravity of Refractory Materials (C 135 - 47),

Warpage of Refractory Brick and Tile (C 154 - 41),

Combined Drying and Firing Shrinkage of Fireclay Plastic Refractories (C 179 - 46),

Workability Index of Fireclay Plastic Refractories (C 181 - 47),

Thermal Conductivity of Insulating Fire Brick (C 182 - 47),

Bonding Strength of Air-Setting Refractory Mortar (Wet Type) (C 198 - 47),

Refractoriness of Air-Setting Refractory Mortar (Wet Type) (C 199 - 47),

Thermal Conductivity of Refractories (C 201 - 47),

Thermal Conductivity of Fireclay Refractories (C 202 - 47), and

Reheat Change of Insulating Fire Brick (C 210 - 46).

Standard Specifications for:

Ground Fire Clay as a Mortar for Laying-Up Fireclay Brick (C 105 - 47),

Fireclay Plastic Refractories for Boiler and Incinerator Services (C 176 - 47), and

Air-Setting Refractory Mortar (Wet Type) for Boiler and Incinerator Services (C 178 - 47).

Standard Symbols for:

Heat Transmission (C 108 - 46).

TENTATIVE CONTINUED WITHOUT REVISION

The committee recommends the continuation without revision of the Tentative Methods for Chemical Analysis of Refractory Materials (C 18 - 56 T).

The recommendations in this report have been submitted to letter ballot of

the committee, the results of which will be reported at the Annual Meeting.[4]

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[3] 1956 Supplement to Book of ASTM Standards, Part 3.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Industrial Survey (R. P. Stevens, chairman) has supplied revisions on the following surveys: "Refractory Service Conditions in By-Product Coke Ovens," and "Refractory Service Conditions in Continuous Glass Furnaces."

Subcommittee II on Research (R. B. Sosman, chairman) is working on the problem of a test for permeability to gases. Tests for refractories under load are also being studied.

Subcommittee III on Tests (S. M. Phelps, chairman) recommended that Method C 269 - 55 T be adopted as a standard test for permanent linear change in firing of castable refractories.

Section A on Load (A. J. Metzger, chairman) is working on a high-temperature load test for mullite refractories.

Section B on Spalling (L. J. Trostel, chairman) is cooperating with the American Iron and Steel Inst. Refractory Committee in the Spalling Test for Blast Furnace Brick. It is also considering the Phelp's hot plate method for determining the resistance to spalling of silica brick.

Section C on Temperature (J. L. Carruthers, chairman) is cooperating with the National Bureau of Standards on the investigation of a standard PCE furnace.

Section D on Precision and Tolerances (Winston Duckworth, chairman) has been reinstituted. The section plans a statistical study of factors influencing strength of certain nonrectilinear shape, and a restudy of present size tolerances of brick specifications.

Section E on Chemical Analysis (H. A. Heiligman, chairman).—The two new standard samples for silica brick are now available at the National Bureau of Standards. They are also arranging for a new supply of crushed chrome ore for standard sample No. 103 which has been exhausted.

The Task Force on Hydration (G. R. Eusner, chairman) has developed procedures for determining the resistance to hydration of basic brick, magnesite grains and dolomite grains.

Section F on Refractory Insulation (W. L. Stafford, chairman) is working on a Class 30 type of insulating brick as well as a specification for insulating fire brick.

Section G on Porosity and Permanent Volume Change (L. C. Hewitt, chairman) is making a study of a standard method using mercury displacement instead of water for materials which hydrate; also a method for determining the size and bulk density of insulating fire brick.

Section H on Mortars, Plastics and Castables (G. H. Anthony, chairman) is making a study of the new T.R.I. castable consistency apparatus.

Section J on Carbon Monoxide Disintegration (K. A. Baab, chairman) is making further studies on the 1957 revisions for C 288 - 56.

Subcommittee IV on Heat Transfer (C. L. Norton, chairman) is working on a test for the thermal conductivity of plastic refractories. Tests for carbon refractories are being investigated.

Subcommittee VI on Nomenclature (A. W. Allen, chairman) is making a study of those terms in the Glossary that need defining and four definitions of various classes of carbon refractories.

Subcommittee VII on Specifications (J. D. Sullivan, chairman) is cooperating with the *ad hoc* committee which is making a study of pouring pit refractories. The *ad hoc* Subcommittee on Pouring Pit Refractories has submitted a proposed specification for study by the main committee.

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[4] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

Subcommittee VIII on Editorial (W. R. Kerr, chairman) has finished the work on the new Eighth Edition of the manual. Committee C-8 is very appreciative of the work done.

Subcommittee IX on Classifications (J. A. Kayser, chairman) has submitted a tentative classification of silica brick. This committee is also working on a proposed classification of raw dolomite, calcine dolomite and dead burned dolomite.

Subcommittee XI on Special Refractories (A. Maupin, chairman) is continuing the study of the classification of mullite refractories.

Subcommittee XII on Carbon Refractories (E. B. Snyder, chairman) has proposed a reheat test for carbon refractories; it also has submitted a proposed nomenclature of carbon brick. Tests on thermal conductivity, alkali attack, and permeability of carbon brick are under study.

Subcommittee XV on Basic Granular Refractories (J. J. Hazel, chairman) is cooperating with various steel companies and suppliers of dead burned dolomite in an effort to develop suitable tests to evaluate this type of refractory material.

This report has been submitted to letter ballot of the committee, which consists of 46 members; 46 members returned their ballot, of whom 45 have voted affirmatively and 1 negatively.

Respectfully submitted on behalf of the committee,

J. J. HAZEL,
Chairman.

L. J. TROSTEL,
Secretary.

REPORT OF COMMITTEE C-9

ON

CONCRETE AND CONCRETE AGGREGATES*

Committee C-9 on Concrete and Concrete Aggregates held two meetings during the year: on June 19, 1957, in Atlantic City, N.J., during the Annual Meeting of the Society, and on December 6, 1957, at Fortin de las Flores, Veracruz, Mexico.

The committee adopted memorials to Lee W. Walter who died on August 7, 1956 and who had been a member of the committee since 1916 and later an honorary member, and to Henry L. Kennedy, a member of the committee for 17 years and at the time of his death, on September 10, 1957, its vice-chairman.

The committee elected I. L. Tyler as Vice-Chairman and E. W. Bauman as a member of the Executive Subcommittee to fill vacancies.

The Sanford E. Thompson Award, established by the committee in 1938 in honor of its first chairman, is made for a paper of outstanding merit on concrete aggregates presented before the Society. The award this year is being made to Tien S. Chang and Clyde E. Kesler of the University of Illinois for their paper entitled "Correlation of Sonic Properties of Concrete with Creep and Relaxation," presented at the Fifty-ninth Annual Meeting of the Society, June 1956.

The officers elected for the ensuing term of two years are as follows:

Chairman, W. H. Price.
Vice-Chairman, I. L. Tyler.
Secretary, Bryant Mather.

NEW TENTATIVE

The committee recommends for publication as tentative the proposed Tentative Method of Test for Abrasion Resistance of Concrete, as appended hereto.[1]

REVISIONS OF TENTATIVES

The committee recommends revisions as indicated of the following tentatives and their continuation as tentative:

Tentative Method of Test for Fundamental Transverse, Longitudinal, and Torsional Frequencies of Concrete Specimens (C 215 – 55 T):[2]

Section 1.—Add a new Paragraph (*b*) to read as follows, lettering the present paragraph as (*a*):

> (*b*) The method is intended primarily for detecting significant changes in the dynamic modulus of elasticity of laboratory or field test specimens that are undergoing exposure to weathering or other types of potentially deteriorating influences. It may be used to assess the uniformity of field concrete but it should not be considered as an index of compressive or flexural strength nor as an adequate test for establishing the compliance of the modulus of elasticity of field concrete with that assumed in design. Comparison of results from specimens of different sizes or shapes should be made with caution since different computed values for the dynamic modulus of elasticity may result from widely different resonant frequencies of specimens of different sizes and shapes of the same concrete.

Section 2(*a*).—Add a Note to read as follows:

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* Sixty-first Annual Meeting of the Society, June 22–27, 1958.

[1] The new tentative appears in the 1958 Book of ASTM Standards, Part 4.

[2] 1955 Book of ASTM Standards, Part 3.

NOTE.—It is recommended that the calibration of the variable frequency audio oscillator be checked periodically against signals transmitted by the National Bureau of Standards radio station WWV, or against suitable electronic equipment such as a frequency counter, the calibration of which has been previously checked and found to be adequate.

Tentative Specifications for Packaged, Dry, Combined Materials for Mortar and Concrete (C 387 - 56 T):[3] Revise as appended hereto.[4]

Tentative Specification for Raw or Calcined Natural Pozzolans for Use as an Admixture in Portland Cement Concrete (C 402 - 57 T):[5]

Section 8(c).—Change the second sentence to read as follows: "The portland cement used in the control mix shall meet the requirements of the Specifications for Portland Cement (ASTM Designation: C 150) and shall be the type and, if available, the brand of cement to be used in the work."

REVISION OF STANDARDS AND REVERSION TO TENTATIVE

The committee recommends revisions as indicated of the following standards and their reversion to tentative:

Standard Method of Test for Measuring Mortar-Making Properties of Fine Aggregate (C 87 - 52):[2]

Section 1.—Delete the last sentence.

Section 2.—Add to the end of the first sentence the words "unless otherwise specified."

Standard Method of Testing Air-Entraining Admixtures for Concrete (C 233 - 55):[2]

Section 7.—At the end of Paragraphs (*b*) and (*e*) change "(Note 1)" to read "(Notes 1 and 2)."

Delete the present Note 2 at the end of the section and replace with a new Note to read as follows:

NOTE 2.—Applicable only when specifically required by the purchaser for use in structures where flexural strength, bond strength, or volume change may be of critical importance.

Standard Specifications for Waterproof Paper for Curing Concrete (C 171 - 56):[3]

Section 4.—Change to read as follows:

4. The paper, when tested in accordance with the Method of Test for Wet Tensile Breaking Strength of Paper and Paper Products (ASTM Designation: D 829), shall conform to the following requirements:

	Direction	
	Machine	Cross
Wet tensile breaking strength, lb per in. of width, min.	30	15

Standard Specifications for Air-Entraining Admixtures for Concrete (C 260 - 54):[2]

Section 3.—Delete the first three lines, from the words "Unless other" through the words "contracting parties."

Paragraphs (*c*), (*e*), *and* (*f*).—Add the parenthetical phrase "(Note 1)" at the end of each of the paragraphs.

New Note.—Add a new Note 1 at the end of Section 3 to read as follows:

NOTE 1.—Applicable only when specifically required by the purchaser for use in structures where flexural strength, bond strength, or volume change may be of critical importance.

ADOPTION OF TENTATIVE AS STANDARD WITHOUT REVISION

The committee recommends that the Tentative Method of Test for Air Content of Freshly Mixed Concrete by the Volumetric Method (**C 173 - 55 T**)[2] be approved for reference to letter ballot of the Society for adoption as standard without revision.

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[3] 1956 Supplement to Book of ASTM Standards, Part 3.

[4] The revised tentatives and standards appear in the 1958 Book of ASTM Standards, Part 4.

[5] 1957 Supplement to Book of ASTM Standards, Part 3.

ADOPTION OF TENTATIVES AS STANDARD WITH REVISION

The committee recommends that the following tentatives be approved for reference to letter ballot of the Society for adoption as standard with revisions as indicated:

Tentative Specifications for Ready-Mixed Concrete (C 94 - 55 T):[2]

Section 3(a)(1).—Delete this paragraph and replace with the following:

(*1*) *Cement.*—Cement shall conform to the Standard Specifications for Portland Cement (ASTM Designation: C 150), the Standard Specifications for Air-Entraining Portland Cement (ASTM Designation: C 175) or the Tentative Specifications for Portland Blast-Furnace Slag Cement (ASTM Designation: C 205). These different cements will produce concretes of different properties and should not be used interchangeably. The purchaser should specify the type or types required, but if no type is specified, the requirements of type I as prescribed in Specifications C 150 shall apply.

Section 6(a).—Change the first sentence to read: "Cement shall be measured by weight, or if permitted by the purchaser, in bags of standard weight (Note 4)."

New Note.—Add a new Note 4 at the end of Section 6(*a*) to read: "NOTE 4.—In the United States the standard weight is 94 lb."

Section 9.—Change Paragraph (*e*) to read as follows:

(*e*) When the concrete is mixed in a truck mixer loaded to its maximum capacity, the number of revolutions of the drum or blades at mixing speed shall be not less than 70 nor more than 100. If the batch is at least ½ cu yd less than the maximum capacity, the number of revolutions at mixing speed may be reduced to not less than 50. All revolutions after 100 shall be at agitating speed.

Change the first sentence of Paragraph (*g*) to read as follows:

(*g*) When a truck mixer or agitator is used for transporting concrete, the concrete shall be delivered to the site of the work and discharge shall be completed within 1½ hr, or before the drum has been revolved 300 revolutions, whichever comes first, after the introduction of the mixing water to the cement and aggregates, or the introduction of the cement to the aggregates, unless a longer time is specifically authorized by the purchaser.

Section 13.—Change the last sentence to read: "In the event of a second failure, the concrete shall be considered to have failed to meet the requirements of the specification."

Section 15.—Change Paragraph (*a*) to read as follows:

15. (*a*) When strength is used as a basis for acceptance of concrete, standard test specimens shall be made in accordance with the Standard Method of Making and Curing Concrete Compression and Flexure Test Specimens in the Field (ASTM Designation: C 31). The specimens shall be cured under standard moisture and temperature conditions in accordance with Section 7 (*a*) and (*b*) of Method C 31 (see Section 18). Strength tests shall be made frequently by the purchaser and, in general, not less frequently than one strength test for each 50 loads of each class of concrete, except that in no case shall a given class of concrete be represented by less than three tests.

Change Paragraph (*d*) to read as follows:

(*d*) To conform to the requirements of these specifications, the average of all of the strength tests (see Section 15 (*b*)) representing each class of concrete, as well as the average of any five consecutive strength tests representing each class of concrete, shall be equal to or greater than the specified strength, and not more than one test in ten shall have an average value less than 90 per cent of the specified strength.

Section 18(a).—Change to read as follows:

(*a*) *Compression Test Specimens.*—Method of Making and Curing Concrete Compression and Flexure Test Specimens in the Field (ASTM Designation: C 31) using standard moist curing in accordance with Section 7(*a*) and (*b*) of Method C 31.

Tentative Method of Test for Bleeding of Concrete (C 232 - 56 T):[3]

Section 3(b).—Change the first sentence to read: "Fill the container with the concrete in accordance with Section 5 of the Method of Test for Weight Per Cubic Foot, Yield and Air Content (Gravimetric) of Concrete (ASTM Designation: C 138), except that the container shall be filled to a height of $10 \pm \frac{1}{8}$ in."

ADOPTION OF TENTATIVE REVISIONS AS STANDARD

The committee recommends that the tentative revisions of the Standard Definitions of Terms Relating to Concrete and Concrete Aggregates (**C 125 – 56**)[2] issued in June 1955 be approved for reference to letter ballot of the Society for adoption as standard.

REVISION OF STANDARDS, IMMEDIATE ADOPTION

The committee recommends for immediate adoption revisions as indicated of the following standards, and accordingly asks for the necessary nine-tenths affirmative vote at the Annual Meeting in order that the revisions may be referred to letter ballot of the Society.

Standard Method of Test for Slump of Portland-Cement Concrete (C 143 – 52):[2] Revise as appended hereto.[4]

Standard Specifications for Liquid Membrane-Forming Compounds for Curing Concrete (C 309 – 56):[2]

Section 2.—Change the last sentence of Paragraph (*c*) to read: "When applied to concrete at the specified rate of application, the compound shall exhibit a daylight reflectance of not less than 60 per cent of that of magnesium oxide."

In Paragraph (*d*) delete the words "after exposure to sunlight for 3 days" and "apparent" from the last sentence.

Section 10(b).—Delete the word "apparent" from the first line.

REAPPROVAL OF STANDARDS

The committee recommends the reapproval of the following standards which have stood for six or more years without revision.

Standard Methods of Test for:

Voids in Aggregate for Concrete (**C 30 – 37**),
Surface Moisture in Fine Aggregate (**C 70 – 47**),
Cement Content of Hardened Portland Cement Concrete (**C 85 – 54**),
Amount of Material Finer than No. 200 Sieve in Aggregate (**C 117 – 49**),
Flow of Portland Cement Concrete by Use of the Flow Table (**C 124 – 39**),
Specific Gravity and Absorption of Coarse Aggregate (**C 127 – 42**),
Sieve Analysis of Fine and Coarse Aggregates (**C 136 – 46**), and
Weight Per Cubic Foot, Yield, and Air Content (Gravimetric) of Concrete (**C 138 – 44**).

Standard Method of:

Sampling Fresh Concrete (**C 172 – 54**), and
Measuring Length of Drilled Concrete Cores (**C 174 – 49**).

TENTATIVES CONTINUED WITHOUT REVISION

The committee recommends the continuation without revision of the following tentatives:

Tentative Method of Test for:

Soundness of Aggregates by Use of Sodium Sulfate or Magnesium Sulfate (**C 88 – 56 T**),
Water Retention Efficiency of Liquid Membrane-Forming Compounds and Impermeable Sheet Materials for Curing Concrete (**C 156 – 55 T**),
Volume Change of Cement Mortar and Concrete (**C 157 – 54 T**),
Potential Alkali Reactivity of Cement-Aggregate Combinations (**C 227 – 52 T**) (NOTE.—During the year, at the request of Committee C-1 on Cement, Committee C-9 assumed jurisdiction over this method),
Resistance of Concrete Specimens to Rapid Freezing and Thawing in Water (**C 290 – 57 T**),
Resistance of Concrete Specimens to Rapid Freezing in Air and Thawing in Water (**C 291 – 57 T**),
Resistance of Concrete Specimens to Slow Freezing and Thawing in Water or Brine (**C 292 – 57 T**),

Resistance of Concrete Specimens to Slow Freezing in Air and Thawing in Water (**C 310 – 57 T**), and

Volume Change of Concrete Products (**C 341 – 54 T**) (NOTE.—By action of the ASTM Board of Directors in May 1958, jurisdiction over this method was reassigned to ASTM Committee C-15 on Manufactured Masonry Units).

Tentative Specifications for:

Lightweight Aggregates for Structural Concrete (**C 330 – 53 T**),

Lightweight Aggregates for Concrete Masonry Units (**C 331 – 53 T**), and

Lightweight Aggregates for Insulating Concrete (**C 332 – 56 T**).

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[6]

ACTIVITIES OF SUBCOMMITTEES

Group I on Administration (I. L. Tyler, chairman).—There are five subgroups within Group I which deal with special phases of Committee C-9 requirements not covered by Groups II and III. The work of Subcommittee I-d (Editorial and Definitions) is closely allied with that of Groups II and III.

Subcommittee I-c submitted recommendations for conferring the Sanford E. Thompson Award, a task requiring a great deal of reading and thought on the part of its members. Subcommittee I-c has carried out its exacting assignment of reviewing standards for their editorial and definition usages. Subcommittee I-b has arranged two sessions to be held during the Annual Meeting in June. Subcommittees I-a and I-e have also been active in their fields of Finance and Membership respectively.

Group II on Research (L. E. Gregg, chairman).—Activities of the Group II or Research Subcommittees have been highly productive during the past year. All seven held at least one meeting and all but one met on both occasions of the meeting of Committee C-9.

In addition to the actions regarding tentatives and standards mentioned previously in the report, proposed tentative methods or recommended practices in three areas of interest were developed for the consideration of Committee C-9. These include a Tentative Method of Test for Static Modulus of Elasticity and Poisson's Ratio of Molded Concrete Specimens in Compression, and a Tentative Method of Test for Creep of Concrete in Compression, both the results of thorough study by Subcommittee II-c. Under the auspices of Subcommittee II-g a Recommended Practice for Determining Air Void Characteristics of Hardened Concrete was brought to virtual completion with only minor details to be resolved before it is submitted to the membership of C-9.

Research projects involving tests among several cooperating laboratories were initiated or extended by Subcommittees II-b and II-g. The former project is based on results of the McPherson Test Road in Kansas where concretes containing various combinations of cements and aggregates have been under observation approximately ten years for evidences of deterioration through cement-aggregate reactions. The intent of the cooperative investigation is to give added significance to results from the standpoint of laboratory tests, particularly microscopic examination for definition of constituents and the products of different types of reactions.

Cooperative research on measurable properties of aggregates which may be related to the characteristics of aggregate pores and believed to be of importance in the resistance of concrete to freezing and thawing was extended by Subcommittee II-g. Data on the aggregates were accumulated and are in the process of analysis. Companion freezing-and-thawing tests on concrete specimens containing the different aggregates are under way, and the correlative data will be available in the near future.

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[6] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

Work similar to cooperative research is being conducted by Subcommittee II-b through two task groups appointed during the past year. One deals with a critical review on alkali-aggregate reaction with respect to tests for pozzolans effective in minimizing the expansion caused by the reaction. The other group is reviewing results of tests from many sources, apparently defining a reaction different from the alkali-aggregate reaction and presently evaluated by the Conrow test.

Changes in scopes of Subcommittee II-a and II-c were proposed, the latter as a means for dealing with a larger number of factors considered influential in elastic and inelastic properties of concrete. In the case of Subcommittee II-a, the intent is to render a broader service to other subcommittees through assistance in the design of experiments for maximum statistical significance, and advice in statistical analysis of their data.

Group III on Specifications and Test Methods (Stanton Walker, chairman).—During the year a critical review was made of the scope of activities of each of the 17 subcommittees and their personnel reviewed. All subcommittees are productively active.

While the accomplishments of the subcommittees are revealed by standards presented, some reference to work under way should be of interest. Brief mention of a very few of the more important projects, without reference to the responsible subcommittee, follows:

1. A comprehensive investigation of the effect on strength of the ratio of length to diameter of test specimen.
2. Consideration of a procedure for determining the cement content of fresh concrete, with one of its principal objectives being measurement of efficiency of concrete mixers.
3. Major revisions in the method of test for abrasion of coarse aggregate by use of the Los Angeles machine (ASTM Designation: C 131) through a joint committee representing Committees C-9 and D-4.
4. A review of the question of mixing time requirements of stationary mixers in relation to their size; the need for such review is indicated by the new types of mixers being manufactured and also by the very much larger sizes being used than when current specification requirements were written.
5. Development of appropriate definitions for initial and final setting times of concrete.
6. Development of a procedure for evaluating the performance of concrete aggregates by freezing and thawing in concrete.

This report has been submitted to letter ballot of the committee, which consists of 164 active members commanding 127 votes plus 6 honorary members having voting privileges; 148 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

W. H. PRICE,
Chairman.

BRYANT MATHER,
Secretary.

REPORT OF COMMITTEE C-11
ON
GYPSUM*

Committee C-11 on Gypsum held two meetings during the year: in Atlantic City, N. J., on June 19 and 20, 1957, and in St. Louis, Mo., on February 10 and 11, 1958.

The committee has brought to the attention of the Administrative Committee on Research a suggested project on Drying of Plaster Prior to Painting for inclusion in the ACR "Challenges in Materials Research."

The committee consists of 37 members, of whom 14 are classified as producers, 8 as consumers, 12 as general interest, and 3 consulting members.

The officers elected for the ensuing term of two years are as follows:

Chairman, G. W. Josephson.
Vice-Chairman, B. W. Nies.
Secretary, R. H. Faber.
Members of Executive Committee:
  Max Barth.
  Theodore I. Coe.
  E. T. Carlson.
  O. H. Storey, Jr.

REVISION OF TENTATIVES

The committee recommends revisions as indicated of the following tentatives and their continuation as tentative:

Tentative Specifications for Precast Reinforced Gypsum Slabs (C 377 – 56 T):[1]

Section 3.—Delete this section, renumbering the subsequent sections accordingly.

Tentative Specifications for Annular Ringed Nails for Gypsum Wallboard (C 380 – 56 T):[1]

Section 1.—Change to read as follows:

1. These specifications cover annular ringed (complete circle) steel wire nails designed for use in the application of $\frac{3}{8}$-in., $\frac{1}{2}$-in., and $\frac{5}{8}$-in. gypsum wallboard.

Section 3(b).—Change to read as follows:

(*b*) The length of nails for application of gypsum wallboard of various thicknesses shall be as follows:

Thickness of Wallboard, in.	Length of Nail, in.
$\frac{3}{8}$	$1\frac{1}{8} \pm \frac{1}{32}$ or $1\frac{1}{4} \pm \frac{1}{32}$
$\frac{1}{2}$	$1\frac{1}{4} \pm \frac{1}{32}$
$\frac{5}{8}$	$1\frac{3}{8} \pm \frac{1}{32}$

REVISIONS OF STANDARDS, IMMEDIATE ADOPTION

The committee recommends for immediate adoption revisions as indicated of the following standards, and accordingly asks for the necessary nine-tenths affirmative vote at the Annual Meeting in order that the revisions may be referred to letter ballot of the Society:

Standard Specifications for Gypsum Plasters (C 28 – 55):[2]

Section 6.—Change to read, "Gypsum ready-mixed plaster shall have a compressive strength of not less than 400 psi."

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* Sixty-first Annual Meeting of the Society, June 22–27, 1958.

[1] 1956 Supplement to Book of ASTM Standards, Part 3.

[2] 1955 Book of ASTM Standards, Part 3.

Section 22.—Change to read as follows:

22. The chemical analysis and physical properties of gypsum plaster shall be determined in accordance with the Methods of Testing Gypsum and Gypsum Products (ASTM Designation: C 26), except that the methods for chemical analysis contained therein are not applicable to gypsum ready-mixed plaster, gypsum wood-fibered plaster, or gypsum concrete.

Standard Specifications for Gypsum Wallboard (C 36 – 55):[2]

Section 2.—Delete the last sentence and replace with the following:

Type "X" (special fire retardant) designates gypsum wallboard, complying with this specification, which provides at least: (1) 1-hr fire retardant ratings for $\frac{5}{8}$ in. thick; or (2) $\frac{3}{4}$-hr fire retardant ratings for $\frac{1}{2}$ in. thick gypsum wallboard applied in single layer nailed application on each face of load-bearing wood framing members when tested in accordance with the requirements of Fire Test of Building Constructions and Materials (ASTM Designation: E 119).

Add a Note 1 to this section to read as follows, renumbering subsequent Notes accordingly:

NOTE 1.—Consult manufacturers for independent test data on assembly particulars, materials, and ratings for other types of construction.

Section 5(c).—Change the maximum length from "14 ft" to read "16 ft."

Standard Definitions of Terms Relating to Gypsum (C 11 – 55):[2]

Add the following new definition:

Gypsum Formboard.—A sheet or slab having an incombustible core, essentially gypsum, surfaced on the exposed side with a fungus-resistant paper and on the reverse side with paper suitable to receive poured-in-place gypsum concrete.

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[3]

This report has been submitted to letter ballot of the committee, which consists of 34 voting members; 22 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

G. W. JOSEPHSON,
Chairman.

O. H. STOREY, JR.,
Secretary.

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[3] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

REPORT OF COMMITTEE C-12
ON
MORTARS FOR UNIT MASONRY*

Committee C-12 on Mortars for Unit Masonry held two meetings during the year: on June 20, 1957, in Atlantic City, N. J., and February 13, 1958, in St. Louis, Mo.

A successful symposium on mortars was held in St. Louis on February 13. Several papers were presented to an audience of approximately 100 technical men from the building industry.

The committee consists of 47 members, of whom 8 are classified as consumers, 18 as producers, and 21 as general interest members.

The officers elected for the ensuing term of two years are as follows:

Chairman, R. E. Copeland.
Vice-Chairman, Harry C. Plummer.
Secretary, C. U. Pierson, Jr.

TENTATIVE CONTINUED WITHOUT REVISION

The committee recommends the continuation without revision of the Tentative Specifications for Aggregate for Masonry Mortar (**C 144 – 52 T**).[1]

SUBCOMMITTEE ACTIVITIES

Subcommittee II on Research and Methods of Test (P. L. Rogers, chairman) proposes to coordinate activities with the sponsoring committee on Masonry Cement of Committee C-1 on Cement, toward improved methods of test on masonry mortars.

Subcommittee III on Specifications (H. C. Plummer, chairman) is preparing an entirely new specification for Mortar for Reinforced Masonry to replace the existing specification C 161 T.

Subcommittee IV on Aggregates (D. L. Bloem, chairman) is studying the effect of sieve analysis of sands on the performance of mortars.

This report has been submitted to letter ballot of the committee, which consists of 47 members; 38 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

R. E. COPELAND,
Chairman.

C. U. PIERSON, JR.,
Secretary.

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* Sixty-first Annual Meeting of the Society, June 22–27, 1958.

[1] 1955 Book of ASTM Standards, Part 3.

REPORT OF COMMITTEE C-13

ON

CONCRETE PIPE*

Committee C-13 on Concrete Pipe held one meeting during the year: in Chicago, Ill. on October 29, 1957.

The committee consists of 42 members of whom 18 are classified as producers, 16 as consumers and 8 as general interest members.

A new subcommittee has been organized to study provisions for rubber gaskets in the several specifications where such gaskets are appropriate (C. A. Bluedorn, chairman).

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1957 Annual Meeting, Committee C-13 presented to the Society, through the Administrative Committee on Standards, the recommendation that the Specification for Concrete Drain Tile be published as tentative. This recommendation was accepted by the Standards Committee on March 31, 1958, and the specification has been assigned the designation C 412–58 T.

REVISION OF STANDARD, IMMEDIATE ADOPTION

The committee recommends for immediate adoption revisions as indicated of the Standard Specifications for Concrete Sewer Pipe (**C 14 - 57**)[1] and, accordingly asks for the necessary nine-tenths affirmative vote at the Annual Meeting in order that these revisions may be referred to letter ballot of the Society.

Section 18.—Change to read as follows:

> 18. Sound full sized pipe shall be tested for leakage under the Permeability Test or Hydrostatic Test and the following percentage of pipe for each method: Permeability Test: 2 per cent of the number of pipe of each size included in the order, but in no case less than five specimens. Hydrostatic Test: ½ of 1 per cent of the number of each size included in the order but in no case less than two specimens.

Section 20.—Change Paragraph (*b*) to read as follows:

> (b) Water pressure as measured by a standardized gage attached to the delivery pipe close to the specimen shall be internally applied to the specimen. The pressure shall be increased uniformly to 10 psi and held at this pressure for 10 min.

Table III.—Delete Footnote *b*.

TENTATIVE REVISION OF STANDARD

The committee recommends the following change in the tentative revision[2] issued June, 1954, of the Standard Specification for Concrete Pipe for Irrigation or Drainage (C 118 - 56).[2]

Section 5.—Change the footnote to read "Where alkali or sulfates are present, sulfate resistant cement shall be used in the manufacturing."

TENTATIVES CONTINUED WITHOUT REVISION

The committee recommends that the

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* Sixty-first Annual Meeting of the Society, June 22–27, 1958.

[1] 1957 Supplement to Book of ASTM Standards, Part 3.

[2] 1956 Supplement to Book of ASTM Standards, Part 3.

following tentatives be continued without revision:

Tentative Specification for:

Reinforced Concrete Culvert, Storm Drain and Sewer Pipe (**C 76 - 57 T**),
Reinforced Concrete Low-Head Pressure Pipe (**C 361 - 57 T**), and
Reinforced Concrete Low-Head Internal Pressure Sewer Pipe (**C 362 - 55 T**).

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[3]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee II on Reinforced Sewer and Culvert Pipe (C. M. Adams, chairman) will study areas of possible change in the new Tentative Specification for Reinforced Concrete Culvert, Storm Drain and Sewer Pipe (C 76 - 57 T).

Subcommittee III on Irrigation and Drain Pipe (A. F. Pillsbury, chairman) will continue work on recommended changes to the Specification for Non-Reinforced Concrete Pipe for Irrigation and Drainage (C 118).

Subcommittee V on Low Head Internal Pressure Sewer Pipe (M. Butler, chairman) will continue work on recommended changes for the Tentative Specification for Reinforced Concrete Low-Head Internal Pressure Sewer Pipe (C 362 - 55 T).

This report has been submitted to letter ballot of the committee which consists of 42 members; 38 members returned their ballots, of whom 37 have voted affirmatively, and 0 negatively.

Respectfully submitted on behalf of the committee,

R. R. LITEHISER,
Chairman.

HOWARD F. PECKWORTH,
Secretary.

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[3] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

REPORT OF COMMITTEE C-14
ON
GLASS AND GLASS PRODUCTS*

Committee C-14 on Glass and Glass Products held two meetings during the year: on October 18, 1957, in Bedford Springs, Pa., and on April 30, 1958, in Pittsburgh, Pa.

The committee consists of 50 voting members, of whom 16 are classified as producers, 18 as consumers, and 16 as general interest members.

The officers elected for the ensuing term of two years are as follows:

Chairman, L. G. Ghering.
Vice-Chairman, Harold Simpson.
Secretary, F. V. Tooley.

REAPPROVAL OF STANDARDS

The committee recommends the reapproval of the following standards which have stood for six or more years without revision:

Standard Methods of:

Chemical Analysis of Glass Sand (**C 146 – 43**),
Flexure Testing of Glass (**C 158 – 43**),
Internal Pressure Test on Glass Containers (**C 147 – 50**), and
Thermal Shock Test on Glass Containers (**C 149 – 50**).

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[1]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Nomenclature and Definitions (H. H. Holscher, chairman) is reviewing the terms relating to fatigue and stress rupture with Subcommittee 3 on Elastic Strength of Materials of Committee E-1 prior to defining glass terms in this area.

Subcommittee II on Chemical Analysis (F. W. Glaze, chairman) is working on the flame photometric methods to determine soda, lime, and magnesia in glass as well as colorimetric methods of determining magnesia and zirconia in glass.

Subcommittee VII on Glass Containers (R. E. Mould, chairman) has completed an inter-laboratory collaborative program for an alternate method of polariscope examination of glass containers which will be added to the Method of Polariscopic Examination of Glass Containers (C 148).

This report has been submitted to letter ballot of the committee, which consists of 50 members; 35 members returned their ballots, of whom 29 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

L. G. GHERING,
Chairman.

F. V. TOOLEY,
Secretary.

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* Sixty-first Annual Meeting of the Society, June 22–27, 1958.

[1] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

REPORT OF COMMITTEE C-15

ON

MANUFACTURED MASONRY UNITS*

Committee C-15 held two meetings during the year: on June 21, 1957, in Atlantic City, N. J., in conjunction with the Annual Meeting of the Society, and on February 11, 1958, in St. Louis, Mo., during ASTM Committee Week.

The committee consists of 73 members, of whom 36 are classified as producers, 17 as consumers, and 20 as general interest members.

The officers elected for the ensuing term of two years are as follows:

Chairman, J. W. Whittemore.

First Vice-Chairman, P. M. Woodworth.

Second Vice-Chairman, E. F. Wanner.

Secretary, M. H. Allen.

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1957 Annual Meeting, Committee C-15 presented to the Society through the Administrative Committee on Standards the following recommendations:

Tentative Revision of Standard Specifications for:

Facing Brick (**C 216 - 50**), and
Structural Clay Facing Tile (**C 212 - 54**).

Tentative Revision of Standard Methods of:

Sampling and Testing Structural Clay Tile (**C 112 - 50**).

Revision of Tentative Specification for:

Ceramic Glazed Structural Clay Facing Tile, Facing Brick and Solid Masonry Units (**C 126 - 55 T**).

New Tentative Specification for:

Industrial Floor Brick (**C 410 - 57 T**).

These recommendations were accepted by the Standards Committee on November 1, 1957, and they appear in the 1957 Supplement to the Book of ASTM Standards, Part 3.

REVISIONS OF STANDARDS, IMMEDIATE ADOPTION

The committee recommends for immediate adoption revisions as indicated of the following standards, and accordingly asks for the necessary nine-tenths affirmative vote at the Annual Meeting in order that the revisions may be referred to letter ballot of the Society.

Standard Specifications for Sewer Brick (Made from Clay or Shale) (C 32 - 50):[1]

Section 1.—Revise the first sentence to read "These specifications cover brick made from clay or shale and burned, and intended for use in drainage structures for the conveyance of sewage, industrial wastes, and storm water, and for related structures such as manholes."

Revise the Note at the end of the section to read as follows:

NOTE.—Brick of grade NA are suitable for use in catch basins, arches, manholes, and for backing.

Table I.—Add to Footnote *a* the sentence "If the average compressive strength is greater than 8000 psi or the

average water absorption is less than 8.0 per cent after 5-hr boiling, the above requirement shall be waived."

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* Sixty-first Annual Meeting of the Society, June 22–27, 1958.

[1] 1955 Book of ASTM Standards, Part 3.

Section 4(b).—Add to the second sentence "except that this shall not apply to grade NA."

Standard Specifications for Building Brick (Solid Masonry Units Made from Clay or Shale) (C 62 – 57):[2]

Section 2(b).—Add the following sentence "When the grade is not specified, grade MW shall govern."

EDITORIAL CHANGES

The committee recommends editorial changes as indicated in the following standards:

Standard Specifications for Building Brick (Solid Masonry Units Made from Clay or Shale) (C 62 – 57):[2]

Section 1(a).—Add the following to the end of the first sentence: "See Paragraph 2(*a*))."

Section 2(g).—Reletter as 2(*a*), move to the beginning of the section, and reletter the subsequent paragraphs accordingly. Capitalize the words "Specifications for facing brick," delete period, and add "(ASTM Designation: C 216)."

Explanatory Note A.—Compressive Strength.—Replace the last sentence with the following:

> Allowable working stresses for brick masonry laid with units having several compressive strengths in several types of mortars are provided in American Standard Building Code Requirements for Masonry, ASA, A41.3.

Explanatory Note A.—Durability.—In the first sentence of the second paragraph replace the words "of the same raw material" with the words "from the same clay deposits."

Explanatory Note B.—Initial Rate of Suction.—Throughout the note change "20 g per min" to read "20 g per min per 30 sq in."

Standard Methods of Sampling and Testing Brick (C 67 – 57):[2]

Section 6.—Add the sentence "Refer to Section 15(*a*) for the definition of a dry specimen."

Section 9.—Add to this section "Brick shall be tested dry. Refer to Paragraph 15(*a*) for the definition of a dry specimen."

Standard Specifications for Facing Brick (Solid Masonry Units Made from Clay or Shale) (C 216 – 57):[2]

Explanatory Note 5.—Wherever the term "20 g per min" appears, change to read "20 g per min per 30 sq in."

These recommendations have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[3]

This report has been submitted to letter ballot of the committee, which consists of 73 members; 58 members have returned their ballots, of whom 57 have voted affirmatively and 1 negatively.

Respectfully submitted on behalf of the committee,

J. W. WHITTEMORE,
Chairman.

M. H. ALLEN,
Secretary.

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[2] 1957 Supplement to Book of ASTM Standards, Part 3.

[3] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee C-15 presented to the Society through the Administrative Committee on Standards the following recommendations:

New Tentative Method of:

Determining the Moisture Condition of Hardened Concrete by the Relative Humidity Method (C 427 – 58 T), and
Determining the Drying Shrinkage of Concrete Block (C 426 – 58 T).

Revision of Tentative Specification for:

Ceramic Glazed Structural Clay Facing Tile, Facing Brick, and Solid Masonry Units (C 126 – 55 T).

Tentative Revision of Standard Specifications for:

Facing Brick (C 216 – 57).

These recommendations were accepted by the Standards Committee on September 9, 1958, and the new and revised tentatives and tentative revision of Standard C 216 appear in the 1958 Book of ASTM Standards, Part 5.

REPORT OF COMMITTEE C-16

ON

THERMAL INSULATING MATERIALS*

Committee C-16 on Thermal Insulating Materials and its subcommittees held two meetings during the year: at Ottawa, Canada, on October 7 to 10, 1957, and at Hollywood, Fla., on March 5 to 7, 1958.

The committee consists of 98 voting members, 26 non-voting members, 8 associate members and 4 honorary members. Of the voting membership 49 are classified as producers, 26 as consumers, and 23 as general interest members.

The officers elected for the ensuing term of two years are as follows:

Chairman, W. L. Gantz.
Vice-Chairman, W. C. Lewis.
Vice-Chairman, C. B. Bradley.
Secretary, J. M. High.
Assistant Secretary, J. R. Allen.
Assistant Secretary, Membership, E. C. Shuman.
Member-at-Large to the Executive Committee, W. L. Scott.

During the year the committee was unfortunate in losing through death the association and friendship of R. K. Thulman who had been a member for a number of years.

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1958 Annual Meeting, Committee C-16 presented to the Society through the Administrative Committee on Standards a proposed Tentative Method of Test for Hot Surface Performance of High-Temperature Thermal Insulations. The tentative method was accepted by the Standards Committee on March 31, 1958 and it will appear in the 1958 Book of ASTM Standards, Part 3, bearing the designation C 411 – 58 T. The method is appended hereto.[1]

NEW TENTATIVES

The committee recommends for publication as tentative the following methods as appended hereto:[1]

Tentative Method of Test for:

Mechanical Stability of Preformed Thermal Insulation by Tumbling,
Making and Curing Test Specimens of Mastic Thermal Insulation Coatings,
Thermal Conductivity of Insulating Materials at Low Temperatures by Means of the Wilkes Calorimeter.

ADOPTION OF TENTATIVES AS STANDARD WITHOUT REVISION

The committee recommends that the following tentatives be approved for reference to letter ballot of the Society for adoption as standard without revision:

Tentative Specifications for:

Molded Cork Pipe Insulation for Low Temperatures (**C 339 – 55 T**), and
Cellular Glass Thermal Insulation for Pipes (**C 381 – 56 T**).

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* Sixty-first Annual Meeting of the Society, June 22–27, 1958.

[1] The new tentatives appear in the 1958 Book of ASTM Standards, Part 5.

Tentative Methods of Test for:

Breaking Strength and Calculated Flexural Strength of Preformed Block-Type Thermal Insulation (**C 203 – 55 T**), and

Wet Adhesion of Thermal Insulating Cements to Metal (**C 383 - 56 T**).

REAPPROVAL OF STANDARDS

The committee recommends the reapproval of the following standards, which have stood for six or more years without revision:

Specifications for:

Mineral Wool Thermal Insulating Cement (**C 195 – 48**).

Methods of Test for:

Storage Density of Thermal Insulating Cement (**C 164 – 52**),

Covering Capacity and Volume Change Upon Drying of Thermal Insulating Cement (**C 166 – 52**),

Thickness and Density of Blanket- or Batt-Type Thermal Insulating Materials (**C 167 – 50**), and

Thermal Conductivity of Materials by Means of the Guarded Hot Plate (**C 177 – 45**).

Symbols for:

Heat Transmission (**C 108 – 46**).

TENTATIVES CONTINUED WITHOUT REVISION

The committee recommends the continuation without revision of the following tentatives:

Tentative Specifications for:

Structural Insulating Board Made from Vegetable Fibers (**C 208 – 57 T**),

Mineral Wool Batt Insulation (Industrial Type) (**C 262 – 55 T**),

Mineral Wool Felt Insulation (Industrial Type) for Low Temperatures (**C 264 – 55 T**),

Mineral Wool Molded-Type Pipe Insulation for Elevated Temperatures (**C 281 – 52 T**),

Mineral Wool Molded-Type Pipe Insulation for Low Temperatures (**C 300 – 52 T**),

Diatomaceous Earth Block-Type Thermal Insulation (**C 333 – 54 T**),

Diatomaceous Earth Thermal Insulation for Pipes (**C 334 – 54 T**),

Calcium Silicate Block-Type Thermal Insulation (**C 344 – 54 T**),

Calcium Silicate Thermal Insulation for Pipes (**C 345 – 54 T**),

Mineral Wool Block or Board Thermal Insulation for Low Temperatures (**C 378 – 56 T**), and

Mineral Wool Felt Insulation (Industrial Type) for Elevated Temperatures (**C 382 – 56 T**).

Tentative Methods of Test for:

Thermal Conductance and Transmittance of Built-up Sections by Means of the Guarded Hot Box (**C 236 – 54 T**),

Thermal Conductivity of Pipe Insulation for Pipes (**C 335 – 54 T**),

Mean Specific Heat of Thermal Insulation (**C 351 – 54 T**),

Water Vapor Transmission of Materials Used in Building Construction (**C 355 – 54 T**), and

Linear Shrinkage of Preformed High-Temperature Thermal Insulation Subjected to Soaking Heat (**C 356 – 55 T**).

Tentative Methods of Testing:

Structural Insulating Board Made from Vegetable Fibers (**C 209 – 57 T**).

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[2]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee T-VI on Thermal Conductivity (Dr. H. E. Robinson, chairman) plans a study on the revision of the Standard Method of Test for Thermal Conductivity of Materials by Means of the Guarded Hot Plate (C 177 – 45). It also intends to promote in all possible ways development of calibrated thermal conductivity specimens for use in furthering the agreement and reliability of guarded hot plate measurements of thermal conductivity at high temperatures. The committee is also exploring methods of measuring thermal conductivity at temperatures above 1500 F.

Subcommittee S-VIII on General Standards (H. P. Hoopes, chairman), heretofore titled Dimensional Standards, will develop standards on such items related to thermal insulation as do not fit properly or are not assigned to other existing subcommittees such as fire tests, handleability, sample plans, etc.

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[2] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

Subcommittee T-IX on Vapor Transmission (F. A. Joy, chairman). The study of prevalent errors in the application of Method of Test for Measuring Water Vapor Transmission of Materials in Sheet Form (E 96 - 53 T) has been made. Publication of these results in the ASTM BULLETIN has been approved. The study of allowable moisture gain by the desiccant in the Methods of Test for Water Vapor Transmission of Materials Used in Building Construction (C 355 - 54 T) is continuing. Future plans include: (1) round-robin tests to establish the reliability of C 355 - 54 T; (2) approval of standard specimens for checking water vapor transmission test procedures, and (3) development of research programs that merit support by Committee C-16 or the Society.

Subcommittee T-X on Physical Properties (Other than Permeance) of Coatings Accessory to Thermal Insulation (R. A. Johnson, chairman) has been conducting round-robin tests on fire resistance on thermal insulation coatings. Plans for future development include round-robin testing on weathering, adhesion, light stability, thermal stability, and flexibility of thermal insulation coatings.

This report has been submitted to letter ballot of the committee, which consists of 98 voting members; 80 members returned their ballots, of whom 76 voted affirmatively and 1 negatively.

Respectfully submitted on behalf of the committee,

W. L. GANTZ,
Chairman.

J. M. HIGH,
Secretary.

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee C-16 presented to the Society through the Administrative Committee revisions of the Tentative Specification for Mineral Wool Molded-Type Pipe Insulation for Elevated Temperatures (C 281 - 52 T). The revisions were accepted by the Standards Committee on September 9, 1958, and the revised specifications appear in the 1958 Book of ASTM Standards, Part 5.

REPORT OF COMMITTEE C-18

ON

NATURAL BUILDING STONES*

Committee C-18 on Natural Building Stones held one meeting during the year: on April 17, 1958, at Washington, D. C. Meetings of the Advisory Subcommittee were held in Washington, D. C. on October 25, 1957 and on April 17, 1958. A meeting of Subcommittee IV on Specifications, including several of the work groups for specifications, was held concurrently with the general meeting of the committee on April 17, 1958.

The Work Groups for Slate and Granite have completed specifications for these two stones which are referred to later in the report. The Marble Group expects to present a proposal for a specification at a meeting of Committee C-18 to be held later in the year.

The officers elected for the ensuing term of two years are as follows:

Chairman, L. W. Currier.
Vice-Chairman, T. I. Coe.
Secretary, F. S. Eaton.

The committee consists of 22 members, of whom 7 are classified as producers, 9 as consumers, and 6 as general interest members. There are 2 consulting members.

NEW TENTATIVE

The committee recommends for publication as tentative the Specification for Building Granite as appended hereto.[1]

ADOPTION OF TENTATIVES AS STANDARD WITHOUT REVISION

The committee recommends that the following tentatives be approved for reference to letter ballot of the Society for adoption as standard without revision:

Tentative Specification for:

Roofing Slate (**C 406 – 57 T**),[2] and

Tentative Method of Test for:

Weather Resistance of Natural Slate (**C 217 – 56 T**).[3]

REAPPROVAL OF STANDARDS

The committee recommends reapproval of the following standards which have stood for six or more years without revision:

Standard Methods of Test for:

Absorption and Bulk Specific Gravity of Natural Building Stone (**C 97 – 47**),
Modulus of Rupture of Natural Building Stone (**C 99 – 52**),
Water Absorption of Slate (**C 121 – 48**),
Compressive Strength of Natural Building Stone (**C 170 –50**), and
Abrasion Resistance of Stone Subjected to Foot Traffic (**C 241 – 51**).

Standard Definition of Terms Relating to:

Natural Building Stones (**C 119 – 50**).

Standard Methods of:

Flexure Testing of Slate (Modulus of Rupture, Modulus of Elasticity) (**C 120 – 52**).

TENTATIVE CONTINUED WITHOUT REVISION

The committee recommends the continuation without revision of the Tentative Method of Test for Combined Effect of Temperature Cycles and Weak Salt Solutions on Natural Building Stone (**C 218 – 48 T**).

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* Sixty-first Annual Meeting of the Society, June 22–27, 1958.

[1] The new tentative appears in the 1958 Book of ASTM Standards, Part 5.

[2] 1957 Supplement to Book of ASTM Standards, Part 3.

[3] 1956 Supplement to Book of ASTM Standards, Part 3.

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[4]

This report has been submitted to letter ballot of the committee, which consists of 20 members; 15 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

L. W. CURRIER,
Chairman.

F. S. EATON,
Secretary.

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[4] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

REPORT OF COMMITTEE C-19

ON

STRUCTURAL SANDWICH CONSTRUCTIONS*

Committee C-19 on Structural Sandwich Constructions held one meeting during the year: on October 16 and 17, 1957, in Madison, Wis., at the Forest Products Laboratory.

The committee consists of 62 voting members, of whom 33 are classified as producers, 12 as consumers, and 17 as general interest members.

The officers elected for the ensuing term of two years are as follows:

Chairman, T. P. Pajak.
Vice-Chairman, J. P. Reese.
Secretary, J. H. Gibbud.
Membership Secretary, Walter Lovett.

SUBCOMMITTEE ACTIVITIES

Subcommittee I on Mechanical Properties of Basic Materials (Steve Yurenka, chairman) is preparing a tentative method for determining fluid flow in honeycomb cores; it is also investigating the need for modification of present standards and tentatives for the testing of core materials at elevated temperatures. The subcommittee prepared a tentative method for determining impact resistance of sandwich core materials which Committee C-19 expects to submit to the Administrative Committee on Standards.

Subcommittee II on Mechanical Properties of Basic Sandwich Construction (E. W. Kuenzi, chairman).—A flexural creep test for sandwich construction is to be prepared which will be in accord with the new method of flatwise flexure testing. The subcommittee is recommending the use of the climbing drum peel test method for adhesives as developed by Committee D-14 on Adhesives. Two other peel test methods are also being considered as alternate and more rapid methods. Other test methods under consideration deal with impact resistance, nondestructive testing, fatigue testing, and tests for flatness.

Subcommittee III on Permanence, Durability, and Simulated Service (J. H. Gibbud, chairman).—The testing of sandwich panels exposed for three years at State College, Pa., and at Kure Beach, N. C., was completed this spring. Exposure program "Beta" is now in its second year. Panels are to be tested after one, two, and three years' exposure.

A proposed tentative procedure for laboratory aging is under consideration and a task group has been appointed to recommend values and cycles.

Subcommittee IV on Nomenclature and Definitions (R. A. Biggs, chairman).—A compilation of additional definitions from two sources is being reviewed. Selected definitions will be mailed to the membership for comments and action. A task force has been assigned the task of recommending a new title and scope for the Standard Method of Test for Delamination Strength of Honeycomb Type Core Material (C 363 – 57).

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* Sixty-first Annual Meeting of the Society, June 22–27, 1958.

This report has been submitted to letter ballot of the committee, which consists of 62 members; 53 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

GEORGE GERARD,
Chairman.

W. L. EMERSON,
Secretary.

REPORT OF COMMITTEE C-20

ON

ACOUSTICAL MATERIALS*

Committee C-20 on Acoustical Materials held two meetings during the year: at Ottawa, Ontario, Canada, on October 10 and 11, 1957; and on April 17, 1958 in Chicago, Ill.

The committee consists of 67 members, of whom 24 are classified as producers, 13 as consumers, 30 as general interest members. There are 9 consulting members.

The officers elected for the ensuing term of two years are as follows:

Chairman, R. N. Hamme.
Vice-Chairman, H. J. Sabine.
Secretary, Ralph Huntley.

NEW TENTATIVE

The committee recommends for publication as tentative the Method of Test for Sound Absorption of Acoustical Materials in Reverberation Rooms as appended hereto.[1]

ADOPTION OF TENTATIVE AS STANDARD WITHOUT REVISION

The committee recommends that the Method of Test for Impedance and Absorption of Acoustical Materials by the Tube Method (**C 384 - 56 T**)[2] be approved for reference to letter ballot of the Society for adoption as standard without revision.

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[3]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Sound Absorption (H. J. Sabine, chairman).—A first draft of the horn coupler method of measuring the sound absorption of specimens by the tube method, which are larger in area than the tube, has been circulated to the subcommittee as a basis for further development of the method. It was recommended that the description of the Armstrong box method for measuring sound absorption be published in the ASTM BULLETIN as information, and that a test program be undertaken to compare the results among boxes of this type.

Subcommittee II on Flame Resistance (Wallace Waterfall, chairman).—Action on the fourth draft of the Method of Flame Resistance Classification of Acoustical Materials will be held in abeyance until interpretation of the behavior of one material which was rated differently by different laboratories in a round-robin test by the panel method is clarified by the Federal Specification Board. A task group was established to arrange for a series of correlation tests by laboratories employing the tunnel method for flame spread rating of acoustical materials by Tentative Method E 84 - 50 T.

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* Sixty-first Annual Meeting of the Society, June 22–27, 1958.

[1] The new tentative appears in the 1958 Book of ASTM Standards, Part 5.

[2] 1956 Supplement to Book of ASTM Standards, Part 4.

[3] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

Subcommittee III on Maintenance (Peter Chrzanowski, acting chairman).—Preliminary experiments at the National Bureau of Standards on soiling of acoustic materials are nearing completion and will be studied in connection with the proposed cleansability and paintability tests. Investigations of the horn-coupled impedance tube and the Armstrong box method are being followed closely to establish the usefulness of these methods for paintability tests. Proposed methods for a quantitative measurement of the amount of paint applied to specimens of acoustic materials are under study.

Subcommittee IV on Application (L. F. Yerges, chairman).—The specification on acoustical tile adhesives was referred to Committee D-14 on Adhesives for presentation to the Society for publication as tentative. The Task Group on Mechanical Systems met with representatives of National Acoustical Contractors Assn., and accessory manufacturers. The objectives of the task group were outlined and a program established.

Subcommittee V on Basic Physical Properties (W. A. Jack, chairman).—A report of preliminary tests on the facility constructed at the Geiger and Hamme Laboratory for measurement of sound transmission through suspended acoustical ceilings indicated that the method shows promise. The subcommittee is continuing to follow this development with a view to preparation of a test method. The development by General Electric Co. of a revised model of the sphere reflectometer is nearing completion and is under study as a possible method for measurement of light reflectance of acoustical materials.

This report has been submitted to letter ballot of the committee, which consists of 67 voting members; 35 members returned their ballots, of whom 33 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

H. A. LEEDY,
Chairman.

J. W. GARRISON,
Secretary.

REPORT OF COMMITTEE C-21

ON

CERAMIC WHITEWARES AND RELATED PRODUCTS*

Committee C-21 on Ceramic Whitewares and Related Products held two meetings during the year: in Bedford Springs, Pa., on September 26, 1957, and in Pittsburgh, Pa., on April 28, 1958.

The committee consists of 77 voting members, of whom 42 are classified as producers, 10 as consumers, and 25 as general interest members.

The officers elected for the ensuing term of two years are as follows:

Chairman, M. D. Burdick.
Vice-Chairman, W. C. Mohr.
Secretary, G. W. Phelps.

Reorganization of the committee structure, under consideration for two years, has been effected.

A new subcommittee on ceramic tile has been organized as Subcommittee VI.

NEW TENTATIVES

The committee recommends for publication as tentative the Method of Test for Crazing Resistance of Fired Glazed Wirewares by Autoclave Treatment as appended hereto.[1]

REVISIONS OF TENTATIVE

The committee recommends revisions consisting of additional definitions, as appended hereto,[1] of the Tentative Definitions of Terms Relating to Ceramic Whitewares and Related Products (**C 242 – 56 T**).[2]

ADOPTION OF TENTATIVES AS STANDARD WITHOUT REVISION

The committee recommends that the following tentatives be approved for reference to letter ballot of the Society for adoption as standard without revision:

Tentative Methods of Test for:

Compressive (Crushing) Strength of Fired Whiteware Materials (**C 407 – 57 T**),[3] and
Thermal Conductivity of Whiteware Ceramics (**C 408 – 57 T**).[3]

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[4]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Nomenclature (A. S. Watts, chairman) has developed 21 definitions common to the whitewares industry and 16 definitions common to the floor and wall tile industry which are being presented to the Society with this report.

Subcommittee II—Editorial (V. E. Campbell, chairman) is editorially reviewing whiteware standards as they are developed within the committee.

Subcommittee III on Fundamental Properties (W. C. Mohr, chairman) is preparing a method to determine the translucency of fired whiteware materials. Methods for lead solubility, tensile strength, glaze scratch resistance, and impact strength are in preparation. Recommendations have been made to Committee D-9 regarding revisions of Methods of Testing Electrical Porcelain (D 116 - 44) and Methods of Testing Steatite Used for Electrical Insulation (D 667 - 44).

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* Sixty-first Annual Meeting of the Society, June 22–27, 1958.

[1] The new and revised tentatives appear in the 1958 Book of ASTM Standards, Part 5.

[2] 1956 Supplement to Book of ASTM Standards, Part 3.

[3] 1957 Supplement to Book of ASTM Standards, Part 3.

[4] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

The test for crazing resistance of fired glazed whitewares by autoclave treatment, referred to earlier in this report, was prepared by the subcommittee.

Subcommittee IV on Clays (G. W. Phelps, chairman), as part of its program to develop a standard method of particle-size analysis of clays, has presented procedures for sample preparation and hydrometer techniques. These studies were published in the *Journal* of the American Ceramic Society, December, 1957. Standard procedures may be developed from these studies.

Subcommittee V on Nuclear Applications (J. M. Warde, chairman), as part of its program to disseminate to the whitewares industry information concerning nuclear applications of ceramic materials, sponsored, with the cooperation of the American Ceramic Society, a symposium at the Bedford Springs meeting of the Materials & Equipment and White Wares Divisions of the American Ceramic Society.

The subcommittee is developing definitions as well as tests and specifications for whiteware materials used in this field.

Task Group A on Non-Metallic Magnetic Materials (George Economos, chairman) is developing test procedures for memory core materials, microwave ferrites, and magnetic powders. Liaison has been established with other ASTM committees and the American Ceramic Society.

Task Group B on Cermets (T. S. Shevlin, chairman) in cooperation with a special ASTM committee appointed by the Board of Directors on Cermets, prepared a background report in order to delineate the distinctive features of cermets so far as Committee C-21 is concerned. The report dealt with nomenclature and definitions, methods of fabrication, current and potential uses and properties.

This report has been submitted to letter ballot of the committee which consists of 77 voting members; 72 members have returned their ballots, of whom 60 have voted affirmatively and 2 negatively.

Respectfully submitted on behalf of the committee,

M. D. BURDICK,
Chairman.

R. W. CLINE,
Secretary.

REPORT OF COMMITTEE C-22
ON
PORCELAIN ENAMEL*

Committee C-22 On Porcelain Enamel held two meetings during the year: on October 8 and 9, 1957, in Chicago, Ill., and on February 10 and 11, 1958, in Middletown, Ohio, as guests of the Armco Steel Corp.

The committee consists of 48 members, of whom 42 are voting members; 22 are classified as producers, 5 as consumers, and 15 as general interest members.

The officers elected for the ensuing term of two years are as follows:

Chairman, W. N. Harrison.
First Vice-Chairman, D. G. Bennett.
Second Vice-Chairman, E. E. Howe.
Secretary, G. H. Spencer-Strong.

REVISION OF TENTATIVE

The committee recommends revisions as appended hereto[1] of the Tentative Definitions of Terms Relating to Porcelain Enamel (**C 286 - 57 T**).[2]

ADOPTION OF TENTATIVES AS STANDARDS WITHOUT REVISION

The committee recommends that the following tentatives be approved for reference to letter ballot of the Society for adoption as standards without revision:

Tentative Method of Test for Thermal Shock Resistance of Porcelain-Enameled Utensils (**C 385 - 56 T**),[3] and
Tentative Classification of Water Used in Milling of Porcelain Enamel (**C 375 - 55 T**).[4]

TENTATIVES CONTINUED WITHOUT REVISION

The committee recommends continuation without revision of the following tentatives:

Tentative Methods of Test for:

Fusion Flow of Porcelain Enamel Frits—Flow-Button Method (**C 374 - 55 T**),
45-Deg Specular Gloss of Ceramic Materials (**C 346 - 55 T**),
Impact Resistance of Porcelain-Enameled Utensils (**C 284 - 51 T**), and
Warpage of Porcelain-Enameled Flatware (**C 314 - 53 T**).

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[5]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Research (B. J. Sweo, chairman) is continuing work in the evaluation of enameling iron and steel for porcelain enameling and has in progress an investigation of the specification test requirements of the dry process cast iron porcelain enameling industry.

Subcommittee II on Nomenclature (E. E. Howe, chairman) prepared the revision of the Tentative Definitions of

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* Sixty-first Annual Meeting of the Society, June 22–27, 1958.

[1] The revised tentative appears in the 1958 Book of ASTM Standards, Part 5.

[2] 1957 Supplement to Book of ASTM Standards, Part 3.

[3] 1956 Supplement to Book of ASTM Standards, Part 3.

[4] 1955 Book of ASTM Standards, Part 3.

[5] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

Terms Relating to Porcelain Enamel (C 286 – 57 T) referred to earlier in the report. Work on definitions is a continuing one.

Subcommittee III on Education (L. S. O'Bannon, chairman) has continued its contacts with the technical press concerning the work of Committee C-22 and has distributed an outline of the work of the committee to some 300 interested companies. The results of this work are reflected in the increased membership of the committee.

Subcommittee IV on Materials (H. S. Saunders, chairman) is expanding its work in the field of stress measurements in porcelain enamel systems and is responsible for the recommendation that C 375 – 55 T be adopted as standard and that Method C 374 – 55 T be retained as tentative as noted earlier in the report.

Subcommittee V on Finished Products (J. C. Richmond, chairman) has practically completed its work on methods of measurement of the resistance of porcelain enamels to abrasion and has made good progress in the development of a test for measuring the resistance of porcelain enamels on aluminum to spalling. It recommended the adoption of Method C 385 – 56 T as standard and the continuation without revision of Tentative Methods C 346 – 55 T, C 284 – 51 T, and C 314 – 53 T referred to earlier in the report.

This report has been submitted to letter ballot of the committee, which consists of 42 voting members; 34 members returned their ballots, of whom 33 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

W. N. HARRISON,
Chairman.

G. H. SPENCER-STRONG,
Secretary.

REPORT OF COMMITTEE D-1

ON

PAINT, VARNISH, LACQUER, AND RELATED PRODUCTS*

Committee D-1 on Paint, Varnish, Lacquer, and Related Products held two meetings during the year: on June 17 to 19, 1957, in Atlantic City, N. J., in connection with the Annual Meeting of the Society, and in Louisville, Ky., on March 5 to 7, 1958.

At the June, 1957, meeting, S. B. Twiss, Assistant Chief Engineer-Research, Engineering Division, Chrysler Corp., Detroit, Mich., presented a paper on "Electron Microscope Study of Weathered Paint Films."[1] This paper gave the results of a study of surface changes in automotive finishes revealed by the electron microscope using a two-stage replica technique, differences in natural and accelerated weathering effects, and correlation of the type and degree of surface breakdown with pigment properties.

At the March, 1958, meeting, R. C. Ernst, Dean of Chemical Engineering, University of Louisville, presented a most enlightening talk on "The Education of the Technical Men of the Future."

A total of 78 standards have been approved jointly by both the Federation of Paint and Varnish Production Clubs and the ASTM, cooperating through the Joint Federation-ASTM Committee on Paint, Varnish, and Lacquer (W. A. Gloger, chairman). A number of additional ASTM standards and tentatives are being considered by the Federation.

The Committee on Intercommittee Relations, composed of Committee D-1 representatives on other ASTM technical committees, is continuing to maintain close contact with those committees whose activities are of interest to Committee D-1. In the last year, an additional representative of Committee D-1 has been appointed to Committee E-12 on Appearance.

During the year the committee approved an amendment to the Regulations Governing Committee D-1 to make provisions for the designation of the time and place for the committee's Annual Meeting by the Chairman and Secretary.

Vice-Chairman E. W. Fasig, who was elected to Committee D-1 in 1924, was given the ASTM Award of Merit by the Society at the June, 1957, Annual Meeting.

Since the June, 1957, meeting, R. D. Bonney, M. Rea Paul, and G. G. Sward were unanimously elected Honorary Members of Committee D-1.

The officers elected for the ensuing term of two years are as follows:

Chairman, W. T. Pearce.
Vice-Chairman, E. W. Fasig.
Secretary, W. A. Gloger.

AMERICAN STANDARDS

Since the last report, the American Standards Association has approved as American Standard the following methods of test and analysis in their revised forms, under the jurisdiction of Committee D-1:

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* Sixty-first Annual Meeting of the Society, June 22–27, 1958.

[1] *Proceedings*, Am. Soc. Testing Mats., Vol. 57, p. 1337, 1957.

Tentative Methods of:

Chemical Analysis of Dry Mercuric Oxide (**D 284 – 57 T**), and
Test for Tinting Strength of White Pigments (**D 322 – 57 T**).

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1957 Annual Meeting, Committee D-1 presented to the Society through the Administrative Committee on Standards the following recommendations:

Tentative Methods of Test for:

Acetone in Methanol (Methyl Alcohol) (**D 1612 – 58 T**),
Acidity in Lacquer Solvents and Diluents (**D 1613 – 58 T**),
Alkalinity in Acetone (**D 1614 – 58 T**),
Ester Value of Lacquer Solvents and Diluents (**D 1617 – 58 T**),
Copper Corrosion by Mineral Spirits (Copper Strip Test) (**D 1616 – 58 T**),
Melamine Content of Nitrogen Resins (**D 1597 – 58 T**),
Glycerol, Ethylene Glycol and Pentaerythritol in Alkyd Resins (**D 1615 – 58 T**), and
Color Difference Using the "Colormaster" Differential Colorimeter (**D 1536 – 58 T**).

Tentative Method of:

Specifying Color by the Munsell System (**D 1535 – 58 T**).

Revision of Tentative:

Method of Test for Unsaponifiable Matter in Alkyd Resins and Resin Solutions (**D 1397 – 56 T**).

These recommendations are currently under consideration by the Administrative Committee on Standards.[2]

NEW TENTATIVES

The committee recommends for publication as tentative the following specifications and methods of test as appended hereto:[3]

Tentative Specifications for:

Distilled Linseed Fatty Acids,
Distilled Soybean Fatty Acids, and
Dehydrated Castor Acids.

Tentative Methods of Test for:

Effect of Staining Agents on Organic Finishes Used in the Transportation Industry,
Total Iodine Value,
Color Change of White Architectural Enamels,
Color of Transparent Liquids (Gardner Color Scale),
Viscosity by Bubble Time Method,
Performance Tests of Clear Floor Sealers, and
Qualitative Tests for Rosin in Varnishes.

REVISION OF TENTATIVE

The committee recommends revision as follows of the Tentative Method of Test for Total Rosin Acids Content of Coating Vehicles (**D 1469 – 57 T**)[4] and continuation of the method as tentative:

Section 1(b).—Change to read as follows:

> (*b*) This method is primarily designed for material containing 0.5 to 5 per cent of rosin on the non-volatile basis.

Tentative Methods of Testing Varnishes (D 154 – 57 T):[5] Add the Method of Panel Preparation for Measurement of Gloss of Varnishes, as appended hereto,[3] and the Method of Test for Print Resistance of Varnishes, as appended hereto.[3]

TENTATIVE REVISIONS OF STANDARDS

The committee recommends tentative revisions as indicated of the following standards:

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[2] Methods D 1535 and D 1536 were accepted by the Standards Committee on June 18, 1958. The remaining recommendations were accepted on July 24, 1958, with the exception of Methods D 1617 and D 1597 which were accepted on September 9, 1958. The new and revised methods appear in the 1958 Book of ASTM Standards, Part 8.

[3] The new and revised tentatives and standards appear in the 1958 Book of ASTM Standards, Part 8.

[4] 1957 Supplement to Book of ASTM Standards, Part 4.

[5] 1956 Supplement to Book of ASTM Standards, Part 4.

Standard Definitions of Terms Relating to Paint, Varnish, Lacquer and Related Products (D 16 – 57):[4]

New Definition.—Add the following new definition:

Gallon, U. S.—A volume equal to 231 cu in. For paint, varnish, lacquer, and related products this is measured at 25 C (77 F).

REVISION OF STANDARDS AND REVERSION TO TENTATIVES

The committee recommends revisions as indicated of the following standards and their reversion to tentatives:

Standard Specifications for Raw Linseed Oil (D 234 – 55):[6]

Section 2.—Add the following requirement: "Flash point, maximum—250 F."

Section 3.—Add the following sentence: "Flash point shall be determined in accordance with the Method of Test for Flash Point of Drying Oils (ASTM Designation: D 1393)."

Standard Specifications for Boiled Linseed Oil (D 260 – 55):[6] Revise as appended hereto.[3]

Standard Specifications for Copper Phthalocyanine Blue (D 963 – 49):[6] Revise as appended hereto.[3]

ADOPTION OF TENTATIVES AS STANDARD WITHOUT REVISION

The committee recommends that the following tentative specifications and methods of test be approved for reference to letter ballot of the Society for adoption as standard without revision:

Tentative Specifications for:

Degummed Soybean Oil (**D 124 – 56 T**),[5]
Dehydrated Castor Oil (**D 961 – 55 T**),[6] and
Safflower Oil (**D 1392 – 56 T**).[5]

Tentative Methods of Test for:

Distillation Range of Lacquer Solvents and Diluents (**D 1078 – 56 T**),[5]
Nonvolatile Content of Resin Solutions (**D 1259 – 56 T**),[5]
Spectrophotometric Diene Value of Dehydrated Castor Oil and Its Derivatives (**D 1358 – 55 T**),[6]
Fire Retardancy of Paints (Cabinet Method) (**D 1360 – 55 T**),[6]
Fire Retardancy of Paints (Stick and Wick Method) (**D 1361 – 55 T**),[6]
Alcohol Content of Ketones (**D 1362 – 57 T**),[4]
Permanganate Time of Lacquer Solvents and Diluents (**D 1363 – 57 T**),[4]
Water in Lacquer Solvents and Diluents (Fischer Reagent Titration Method) (**D 1364 – 55 T**),[6]
Abrasion Resistance of Clear Floor Coatings (**D 1395 – 56 T**),[5]
Chemical Analysis of Poly (Vinyl Butyral) (**D 1396 – 56 T**),[5]
Fatty Acid Content of Alkyd Resins and Resin Solutions (**D 1398 – 56 T**),[5] and
Unsaponifiable Content of Tricresyl Phosphate (**D 1399 – 56 T**).[5]

Tentative Methods of Testing:

Varnishes (**D 154 – 56**)[5] (Tentative revision of Sections 30, 31, and 32, June, 1956), and
Asphalt Emulsions for Use as Protective Coatings for Metal (**D 1010 – 55 T**),[6] jointly with Committee D-8 on Bituminous Materials for Roofing, Waterproofing, and Related Building or Industrial Uses.

Tentative Methods of Measurement of:

Dry Film Thickness of Non-Metallic Coatings of Paint, Varnish, Lacquer, and Related Products Applied on a Non-Magnetic Base (**D 1400 – 56 T**).[5]

ADOPTION OF TENTATIVES AS STANDARD WITH REVISIONS

The committee recommends that the following tentatives be approved for reference to letter ballot of the Society for adoption as standard with revisions as indicated:

Tentative Specifications for Acetone (**D 329 – 55 T**):[6] Revise as appended hereto.[3]

Tentative Specifications for Methyl Isobutyl Ketone (**D 1153 – 55 T**):[6] Revise as appended hereto.[3]

REVISIONS OF STANDARDS, IMMEDIATE ADOPTION

The committee recommends for immediate adoption revisions as indicated of the following standards, and accordingly asks for the necessary nine-tenths affirmative vote at the Annual Meeting in order that the revisions may be referred to letter ballot of the Society.

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[6] 1955 Book of ASTM Standards, Part 4.

Standard Methods of Testing Drying Oils (D 555 - 54):[6]

Section 34.—Change Paragraph (*a*) to read: "Acetone, ACS Grade."

Section 36.—In Paragraph (*b*) change the seventh sentence to read: "Clamp the tube in a vertical position and allow to stand for 24 hr at 25 ± ½ C."

Standard Methods of Test for Viscosity of Paints, Varnishes, and Lacquers by Ford Viscosity Cup (D 1200 - 54):[6] Revise as appended hereto.[3]

Standard Method of Test for Sieve Analysis of Glass Spheres (D 1214 - 54):[6]

Section 2.—In line 3, change the word "smallest" to "largest."

Section 3.—In line 5, change the word "increasing" to "decreasing."

Section 4.—In line 1, change "riffling" to read "riffle sampling."

Section 5.—In Paragraph (*b*), line 3, change "smallest" to "largest." In Paragraph (*b*), line 7, change "as so" to "so as." In Paragraph (*d*), replace the first sentence with: "Weigh the portion of the sample retained on the sieve to the nearest 0.1 g. Place the material passing through the largest sieve on the sieve with the next smaller opening for the series selected for the sieve analysis." In line 8 of Paragraph (*d*) change "increasing" to "decreasing."

Standard Methods of Sampling and Testing Lacquer Solvents and Diluents (D 268 - 53):[6]

Section 10.—Replace with a reference to the Method of Test for Acidity in Lacquer Solvents and Diluents (ASTM Designation: D 1613).

Section 11(b).—Replace with a reference to the Method of Test for Alkalinity in Acetone (ASTM Designation: D 1614).

Section 12.—Replace with a reference to the Method of Test for Ester Value of Lacquer Solvents and Diluents (ASTM Designation: D 1617).

Standard Specifications for Ethyl Acetate (85 to 88 per cent Grade) (D 302 - 33):[6] Revise as appended hereto.[3]

Standard Specifications for Normal Butyl Acetate (88 to 92 per cent Grade) (D 303 - 40):[6] Revise as appended hereto.[3]

Standard Specifications for Butanol (Normal Butyl Alcohol) (D 304 - 45):[6] Revise as appended hereto.[3]

Standard Specifications for Amyl Acetate (Synthetic) (85 to 88 per cent Grade) (D 318 - 39):[6] Revise as appended hereto.[3]

Standard Specifications for Amyl Alcohol (Synthetic) (D 319 - 40):[6] Revise as appended hereto.[3]

Standard Specifications for Amyl Acetate Made From Fusel Oil (85 to 88 per cent Grade) (D 554 - 39):[6] Revise as appended hereto.[3]

Standard Specifications for Dibutylphthalate (D 608 - 43):[6] Revise as appended hereto.[3]

Standard Specifications for Isopropyl Acetate (D 657 - 44):[6] Revise as appended hereto.[3]

Standard Specifications for Isopropyl Alcohol (D 770 - 46):[6] Revise as appended hereto.[3]

Standard Specifications for Secondary Butyl Acetate (85 to 88 per cent Grade) (D 966 - 52):[6] Revise as appended hereto.[3]

Standard Specifications for Secondary Butyl Alcohol (D 1007 – 52):[6] Revise as appended hereto.[3]

Standard Specifications for Methanol (Methyl Alcohol) (D 1152 – 54):[6] Revise as appended hereto.[3]

WITHDRAWAL OF STANDARD

The committee recommends the withdrawal of the Standard Method of Test for Light Sensitivity of Traffic Paint (**D 712 – 47**) because it is no longer a meaningful test.

REAPPROVAL OF STANDARDS

The committee recommends the reapproval of the following standards which have stood for six or more years without revision:

Standard Method of Test for:

Phthalic Anhydride Content of Alkyd Resins and Resin Solutions (**D 563 – 52**),
Abrasion Resistance of Coatings of Paint, Varnish, Lacquer, and Related Products with the Air Blast Abrasion Tester (**D 658 – 44**),
Evaluating Degree of Settling of Traffic Paint (**D 869 – 48**),
Abrasion Resistance of Coatings of Paint, Varnish, Lacquer, and Related Products by the Falling Sand Method (**D 968 – 51**),
Night Visibility of Traffic Paints (**D 1011 – 52**),
Total Nitrogen in Resins for Surface Coatings (**D 1013 – 52**), and
Total Chlorine in Polyvinyl Chloride Polymers and Copolymers Used for Surface Coatings (**D 1156 – 52**).

Standard Methods of Testing:

Nitrocellulose Clear and Lacquer Enamels (**D 333 – 40**), and
Soluble Nitrocellulose Base Solutions (**D 365 – 39**).

Standard Method for Measurement of:

Dry Film Thickness of Paint, Varnish, Lacquer, and Related Products (**D 1005 – 41**).

Standard Methods of Evaluating:

Degree of Resistance to Rusting Obtained with Paint on Iron or Steel Surfaces (**D 610 – 43**),
Degree of Resistance to Chalking of Exterior Paints of the Linseed Oil Type (**D 659 – 44**),
Degree of Resistance to Checking of Exterior Paints of the Linseed Oil Type (**D 660 – 44**),
Degree of Resistance to Cracking of Exterior Paints of the Linseed Oil Type (**D 661 – 44**),
Degree of Resistance to Erosion of Exterior Paints of the Linseed Oil Type (**D 662 – 44**),
Degree of Resistance to Flaking (Scaling) of Exterior Paints of the Linseed Oil Type (**D 772 – 47**),
Degree of Resistance of Traffic Paint to Abrasion, Erosion, or a Combination of Both, in Road Service Tests (**D 821 – 47**),
Degree of Resistance of Traffic Paints to Bleeding (**D 868 – 48**), and
Degree of Resistance of Traffic Paints to Chipping (**D 913 – 51**).

TENTATIVE CONTINUED WITHOUT REVISION

The committee recommends the continuation without revision of the Tentative Method of Test for No-Smear Time of Traffic Paint (**D 1359 – 55 T**).

EDITORIAL CHANGES

The committee recommends editorial changes as indicated in the following standards:

Standard Methods of Testing Varnishes (D 154 – 56):[5]

New Notes.—At the end of the present procedure for testing color, add a Note referring to the Tentative Method of Test for Color of Transparent Liquids (Gardner Color Scale) as an alternate method, as appended hereto.[3] At the end of the present procedure for testing viscosity, add a Note referring to the Tentative Method of Test for Viscosity by Bubble Time Method as an alternate method.

Standard Method of Test for Elongation of Attached Lacquer Coatings with the Conical Mandrel Test Apparatus (D 522 – 41):[6]

Section 4.—Change the first sentence to read: "At least three replicate specimens shall be tested under conditions of temperature and humidity mutually agreed upon by the purchaser and the seller."

Standard Method of Test for Abrasion Resistance of Coatings of Paint, Varnish, Lacquer, and Related Products with the Air Blast Abrasion Tester (D 658 - 44):[6]

Footnote 4.—Revise to read as follows:

[4] Suitable silicon carbide grain may be obtained by sieving Carborundum Grain No. 180 GG obtainable from the Carborundum Co., Niagara Falls, N. Y., or Crystolon Special Grain 200 mesh No. 5999 obtainable from the Norton Co., Teterboro, N. J. The abrasive should be sieved for a minimum of ½ hr with vigorous agitation, and only that portion which passes through a No. 170 (88-micron) sieve and is retained on a No. 200 (74-micron) sieve should be retained for use in the test. The retained portion of the abrasive is considered satisfactory if a minimum of 85 per cent by weight meets the grain size requirements of 88 to 74 microns. Other types of grades of abrasive may be used when mutually agreed upon by the purchaser and the seller.

Section 7.—In the first sentence and in the Note, the trade name "Carborundum" should be replaced by the more general "silicon carbide grain."

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[7]

This report has been submitted to letter ballot of the committee, which consists of 376 voting members; 204 members returned their ballots, of whom 180 have voted affirmatively and 1 negatively.

Respectfully submitted on behalf of the committee,

W. T. PEARCE,
Chairman.

W. A. GLOGER,
Secretary.

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[7] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

REPORT OF COMMITTEE D-2
ON
PETROLEUM PRODUCTS AND LUBRICANTS*

This report contains the recommendations of the subordinate groups of Committee D-2 on Petroleum Products and Lubricants. The recommendations have been approved by Committee D-2 in accordance with the regulations of the Society and are presented herewith.

The officers elected for the ensuing term of two years are as follows:

Chairman, H. M. Smith.

First Vice-Chairman, R. C. Alden.

Second Vice-Chairman, W. K. Simpson.

Secretary, W. T. Gunn.

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1957 Annual Meeting, Committee D-2 presented to the Society through the Administrative Committee on Standards the recommendation for the revision of Tentative Specifications for Gasoline (D 439 – 56 T). This recommendation was accepted by the Standards Committee on August 26, 1957, and the revised tentative specifications appear in the 1957 Supplement to Book of ASTM Standards, Part 5.

PROPOSED METHODS PUBLISHED AS INFORMATION

The following 15 proposed methods are recommended for publication as information, or for republication as information, as appended[1] or as otherwise indicated:

APPENDIX I. Dilution of Automotive Crankcase Oils (Reduced Pressure Distillation Method).[2]

APPENDIX II. Method of Computing ASTM Butadiene Measurement Tables.

APPENDIX III. Filterability of Aviation Turbine Fuels.

APPENDIX IV. Particulate Matter in Hydrocarbons.

APPENDIX V. Sealing Strength of Paraffin Wax.

APPENDIX VI. Breaking Strength of Petroleum Waxes.

APPENDIX VII. Hydrocarbon Types in Low Olefinic Gasoline by Mass Spectrometry.

APPENDIX VIII. Maximum Fluidity Temperature of Residual Fuel Oil.

APPENDIX IX. Ash Content of Petroleum Oils.

APPENDIX X. Knock Characteristics of Motor Fuels Above 100 Octane Number by the Research Method.

APPENDIX XI. Specifications for Aviation Turbine Fuels.

APPENDIX XII. Thermal Stability of Aviation Turbine Fuels.

APPENDIX XIII. Distillation of Petroleum Products.

APPENDIX XIV. Knock Characteristics of Motor Fuels Above 100 Octane Number by the Motor Method.

APPENDIX XV. "Gasoline Engine Crank case Oil Dilution Tests." A Report of Cooperative Tests by Section Q-1, Technical Committee B, by H. E. Luntz.

APPENDIX TO METHOD D 128, ANALYSIS OF LUBRICATING GREASES: Publish as information as an Appendix to Method D 128, the Optional Method for Analysis of Lubricating Greases.[3]

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* Sixty-first Annual Meeting of the Society, June 22–27, 1958.

[1] The proposed methods appear in the 1958 compilation of ASTM Standards on Petroleum Products and Lubricants.

[2] A report of cooperative tests comparing this proposed method with ASTM Method D 322 appears in Appendix XV.

[3] 1957 Supplement to Book of ASTM Standards, Part 5.

NEW TENTATIVES

The committee recommends for publication as tentative the following seven methods as appended hereto.[4]

Methods for Calculation of Volume and Weight of Benzene, Toluene, and Paraxylene (jointly with Committee D-16),
ASTM Butadiene Measurement Tables,
Zinc in New Lubricating Oils and Lubricating Oil Additives (Polarographic Method),
Vanadium in Navy Special Fuel Oil,
Sulfur in Petroleum Oils by Quartz-Tube Combustion,
Methods for Analysis of Graphites Used as Lubricants, and
High-Temperature Combustion Method for Sulfur in Petroleum Products.

REVISIONS OF TENTATIVES

The committee recommends revisions as indicated of the following 17 tentative methods of test and their continuation as tentative:

D 96 – 57 T,[3] Test for Water and Sediment by Means of the Centrifuge:

Section 4 (a).—Change the first sentence to read "Measure exactly 50 ml of industrial benzene (ASTM Designation D 837),[4] saturated with water at ambient temperature, into the centrifuge tube and add exactly 50 ml of the oil to be tested."

D 322 – 55 T,[5] Test for Dilution of Crankcase Oils: Revise as appended hereto.[4]

D 381 – 57 T,[3] Test for Existent Gum in Fuels by Jet Evaporation:

Section 1.—In the first sentence, change "covers" to read "describes," and change "aviation fuels" to read "aircraft fuel." Delete the second sentence.

Sections 2 and 3.—Interchange the numbers for these sections. Entitle Section 2 "Definition" and Section 3 "Outline of Method." Change to read as follows:

2. *Existent Gum* is the evaporation residue of aircraft fuel or the heptane-insoluble portion of the evaporation residue of motor gasoline.

3. A measured quantity of fuel is evaporated under controlled conditions of temperature and flow of air or steam. For aviation gasoline and aircraft turbine fuel, the resulting residue is weighed and reported as milligrams per 100 ml. For motor gasoline, the residue is extracted with heptane, dried, weighed, and reported as milligrams per 100 ml.

Note 1.—Delete this note and renumber the subsequent notes accordingly.

Section 4 (h).—Change "Crucible[4]" to read "Funnel,[4]" and make the same change in Footnote 4.

Section 8.—In Paragraph (*d*), the second sentence, change "crucible" to read "funnel," and add to the end of the sentence "(see Section 10 (*b*))."

Change Paragraphs (*f*) and (*g*) and add new Paragraphs (*h*), (*i*), (*j*), (*k*) and (*l*) to read as follows:

(*f*) At the end of the heating period, remove the beakers from the bath and allow them to cool to room temperature. Segregate the beakers containing the residues from motor gasolines for finishing as described in Paragraphs (*h*) to (*l*). Treat the remaining beakers as described in Paragraph (*g*).

(*g*) Place the beakers containing the residues from aviation gasolines and aircraft turbine fuels in the cooling vessel in the vicinity of the balance for at least 2 hr. Weigh the beakers in the same manner as described in Paragraph (*c*). Record the weights. Calculate the existent gum as described in Section 9.

(*h*) To each of the beakers containing the residues from motor gasolines segregated as directed in Paragraph (*f*), add 25 ml of normal heptane and swirl gently for 30 sec. Allow the mixture to stand for 10 min. Treat the tare beaker in the same manner.

(*i*) Decant and discard the heptane solution, taking precaution to prevent the loss of any solid residue.

(*j*) Repeat the extraction with a second 25-ml portion of heptane as described in Paragraphs (*h*) and (*i*). Repeat the extraction a third time if the extract is colored.

(*k*) Place the beakers, including the tare, in the gum bath maintained at 320 to 329 F (160 to 165 C) and, without replacing the conical jets, allow the beakers to dry for 5 min.

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[4] The new and revised tentatives and standards appear in the 1958 Book of ASTM Standards, Part 7.

[5] 1955 Book of ASTM Standards, Part 5.

(*l*) At the end of the drying period, remove the beakers from the bath, place them in a cooling vessel, and allow to cool in the vicinity of the balance for at least 2 hr. Weigh the beakers in the same manner as described in Paragraph (*c*). Record the weights. Calculate the existent gum as described in Section 9.

Section 9.—Delete this section.

Section 10. Renumber as 9. Change Paragraphs (*a*) and (*b*) to read as follows:

(*a*) If a double-pan balance is used, calculate the existent gum content as follows:

$$A = 2000\,(B - C)$$

where:

A = existent gum content, mg per 100 ml,
B = weight of sample beaker plus existent gum, in g, and
C = weight of empty sample beaker in g.

(*b*) If a single-pan balance is used, calculate the existent gum content as follows:

$$A = 2000\,(B - C + X - Y)$$

where:

A = existent gum content, mg per 100 ml,
B = uncorrected weight of sample beaker plus existent gum in g,
C = weight of empty sample beaker, in g,
X = original weight of tare beaker, in g, and
Y = final weight of tare beaker, in g.

Section 11.—Renumber as 10(*a*). In lines one and two, delete "or the solvent-washed gum" and ", or both." Add a new Paragraph (*b*) to read as follows:

(*b*) After the numerical value for existent gum, designate by the word "filtered" the fact that extraneous material has been removed as described in Section 8 (*d*).

Section 12.—Renumber as 11 and change to read as follows:

11. *Precision.*—Two determinations on the same sample should not differ from each other by more than the amounts shown in the following Fig. 2.

Figure 1.—Change the caption to read "Apparatus for Determining Existent Gum by Jet Evaporation."

New Figure.—Add a new Fig. 2 as shown in the accompanying Fig. 1.

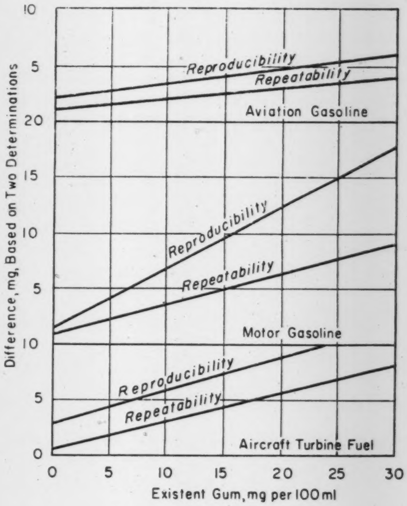

FIG. 1.—Precision.

D 439 – 57 T,[3] Specifications for Gasoline:

Appendix.—In the Section on *Gum*, change the second paragraph to read as follows:

Many motor gasolines are deliberately blended with nonvolatile oils or additives which remain as evaporation residues in the initial evaporation step in the ASTM Test for Existent Gum. This latter method, however, now includes a heptane washing step which removes the heptane soluble material in order to determine the existent gum.

D 524 – 52 T,[5] Test for Carbon Residue of Petroleum Products, Ramsbottom Coking Method:

Section 5 (*b*).—Replace the third, fourth, and fifth sentences with the following:

By means of a hypodermic syringe or the device shown in Fig. 3 introduce into the coking bulb an amount of sample as indicated in

Table I. Make sure that no oil remains on the exterior surface or on the inside of the neck of the bulb. Reweigh the bulb and contents to the nearest milligram. If the sample foams or spatters, repeat the test using the next smaller sample size listed in Table I.

Section 5 (*c*).—Add to the last sentence "and repeat the determination using a smaller sample (Note 12)."

TABLE I.—SAMPLE SIZES.

Ramsbottom Carbon Residue, per cent	Sample Size, g
Less than 6.0	4.0 ± 0.1
6.0 to 14.0	1.0 ± 0.1
14.1 to 20.0	0.5 ± 0.1

Section 8.—Change Paragraphs (*a*) and (*b*) to read as follows:

(*a*) Duplicate results by the same operator should not be considered suspect unless they differ by more than the following amounts:

Ramsbottom Carbon Residue, per cent	Repeatability, per cent of Mean
0.1 to 6.0	see Fig. 4
6.1 to 20.0	10

(*b*) The result submitted by one laboratory should not be considered suspect unless it differs from that of another laboratory by more than the following amounts:

Ramsbottom Carbon Residue, per cent	Reproducibility, per cent of Mean
0.1 to 6.0	see Fig. 5
6.1 to 20.0	15

Footnote 2.—Add a sentence to read as follows:

In 1958, additional data pertaining to precision were incorporated, and the procedure was revised to permit testing samples having high percentages of carbon residue.

D 613 – 56 T,[6] Ignition Quality of Diesel Fuels by the Cetane Method:

Section 6 (*n*).—Change to read as follows:

(*n*) Compression Ratio Adjustment; micrometer set on cylinders of standard bore to read 2.000 in. (10.0 to 1 compression ratio) when the clearance volume is 72 ml to the top face of combustion pickup hole. Clearance volumes for oversize cylinders are shown in Section 304.[3] Volumetric measurements must be made with the piston on top dead center and with the piston over-travel set at 0.015 ± 0.001 in., by using shims between the crankcase and cylinder.

In addition, the following changes are to be made in the supplementary material to Method D 613 which appears in the 1952 ASTM Manual of Engine Test Methods for Rating Fuels.

Supplement I

Section 104.—In descriptive dimensions change second item "Bore, in." to read "Standard Bore, in."

Section 105 (*b*).—Change to read as follows:

(*b*) *Cetane*.—The cylinder, with a detachable head, is made of cast iron, bored and honed, and has a Brinell hardness of 220 ± 20. Cylinders of standard bore are preferred equipment but rebored cylinders up to a maximum of 0.030 in. oversize may be used. The cylinder head is of the variable compression ratio, high turbulence type.

Supplement III:

Section 304 (*c*).—Change to read as follows:

(*c*) *Volume of Water*.—From a clean buret, measure 72 ml of water into the combustion chamber through the combustion indicator hole on cylinders of standard bore, both water and engine being at room temperature. For oversize cylinders of 0.010, 0.020 and 0.030 in., the clearance volumes should be 72.4, 72.8 and 73.2, respectively. Adjust the handwheel accurately to bring the water level to the top face of the indicator hole.

Supplement IV:

Section 410 (*b*).—Change to read as follows:

(*b*) *Checking Cylinders for Wear*.—Cylinders should be checked for wear at intervals of about every 500 hr. Replace the cylinder when the wear becomes 0.006 in., or the taper becomes

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[6] 1957 Supplement to ASTM Manual of Engine Test Methods for Rating Fuels.

0.004 in., or the cylinder is out of round by 0.0025 in. Give particular attention to the wear at the top of the ring travel as it is normally greatest at this point. Cylinders of standard bore are preferred equipment but rebored cylinders up to a maximum of 0.030 in. oversize may be used. In calculating the amount of wear, be sure to use the actual diameter of the standard or the oversize cylinder before placing either in service rather than the nominal standard or oversize diameter. The amount of wear, taper and out of round permitted on oversize cylinders is the same as for cylinders of standard bore.

Table XVIII.—Under Cylinder, change "Bore" to read "Standard Bore," and change replacement limits for bore diameter from "3.256" to "0.006."

D 614 - 56 T,[6] Knock Characteristics of Aviation Fuels by the Aviation Method:

Section 6 (n).—Change to read as follows:

(*n*) *Micrometer Adjustment,* set to read 0.500 in. (5.5 to 1 compression ratio) with the piston at tdc and with a clearance volume of 142 ml to the top face of the thermal plug hole on cylinders of standard bore as measured by either of the tilt procedures described in Sections 302 and 303. Clearance volumes for oversize cylinders are shown in Section 302.

In addition, the following changes are to be made in the supplementary material to Method D 614 which appears in the 1952 ASTM Manual of Engine Test Methods for Rating Fuels.

Supplement I:

Section 104.—Change to read as recommended above for Method D 613.

Section 105 (a).—Add as the second sentence "Cylinders of standard bore are preferred equipment but rebored cylinders up to a maximum of 0.030 in. oversize may be used."

Section 106 (b).—Change the second paragraph to read as follows:

Rings.—Three wedge type compression rings and two wedge type oil control rings are required. Two sets of rings are approved. The preferred set[a] consists of a chromium-plated top compression ring, Waukesha Part No. 105905-C, two plain compression rings, Waukesha Part No. 105905-A, and two narrow-faced oil control rings, Waukesha Part No. 109442. The other set consists of three plain compression rings, Waukesha Part No. 105905-A and two wide-faced oil control rings, Waukesha Part No. 106331. When new, ring-gap clearances are 0.015 to 0.020 in. for the compression rings, and 0.010 to 0.018 in. for the oil rings.

Supplement IV:

Section 410 (b).—Change to read as recommended above for Method D 613.

Table XVIII.—Change to read as recommended above for Method D 613.

D 892 - 46 T,[3] Test for Foaming Characteristics of Lubricating Oils:

Section 3 (a).—In the first sentence, add "shown in Fig. 2," after the words "air-inlet tube." Replace the second sentence with the following:

The cylinder shall have a diameter such that the distance from the inside bottom to the 1000-ml graduation mark is 360 ± 25 mm. It shall be circular at the top (Note 1) and shall be fitted with a rubber stopper having one hole at the center for the air-inlet tube and a second hole off-center for an air-outlet tube. The air-inlet tube shall be adjusted so that, when the rubber stopper is fitted tightly into the cylinder, the diffuser stone (Note 2) just touches the bottom of the cylinder and is approximately at the center of the circular cross-section. Diffuser stones shall meet the following specification when tested in accordance with the method given in the Appendix:

Maximum pore diameter, microns: not greater than 80
Permeability, milliliters of air per minute at pressure of 250 mm water: 3000 to 6000

Note 1.—Change to read as follows:

NOTE 1.—Graduated cylinders with circular tops may be prepared from cylinders with pouring spouts by cutting them off below the spouts.

New Note.—Add a new Note 2 to read as follows, renumber the present Note 2 as Note 3 and change the refer-

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[a] Not effective until January 1, 1959.

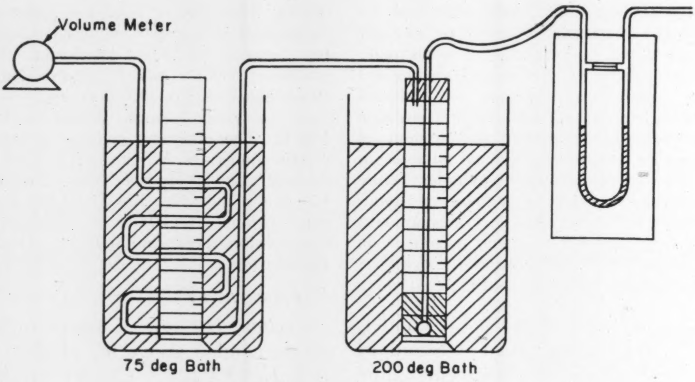
Volume Meter
75 deg Bath
200 deg Bath

FIG. 2. Foaming Test Apparatus.

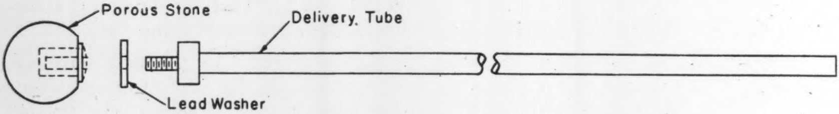
Porous Stone
Delivery Tube
Lead Washer

FIG. 3. Attachment of Diffuser Stones to Air-Inlet Tubes.

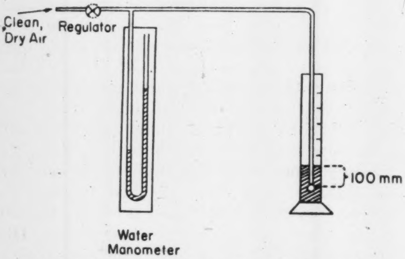
Clean, Dry Air
Regulator
100 mm
Water Manometer

FIG. 4.—Setup for Measuring Maximum Pore Size.

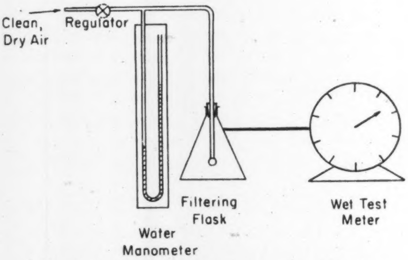
Clean, Dry Air
Regulator
Filtering Flask
Wet Test Meter
Water Manometer

FIG. 5.—Setup for Measuring Permeability.

ences to the present Note 2 to read Note 3.

NOTE 2.—Diffuser stones may be attached to air-inlet tubes by any suitable means. A convenient arrangement is shown in Fig. 2.

Figure 1.—Replace with the accompanying Fig. 2.

New Figures.—Add new Figures 2, 3, and 4 as shown in the accompanying Figs. 3, 4, and 5.

Section 3.—Delete Paragraph (*c*) and reletter the subsequent paragraphs accordingly. Reletter the present Paragraph (*d*) as (*c*) and change to read as follows:

(*c*) *The Air Supply* shall be from a source capable of maintaining an air flow rate of 94 ± 5 ml per min through the gas diffuser stone. The air shall be passed through a drying tower 300 mm in height packed as follows: Just above the constriction place a 20-mm layer of cotton, then a 110-mm layer of desiccant, a 40-mm layer of indicating desiccant, a 30-mm layer of desiccant and a 20-mm layer of cotton. The cotton serves to hold the desiccant in place

Refill the tower when the indicating desiccant begins to show presence of moisture. Any type of flowmeter sensitive to the required tolerances may be used to measure air flow (Note 4).

The total volume of air leaving the foaming test apparatus shall be measured by a volume measuring device (Note 5) capable of accurately measuring gas volumes of about 470 ml. The air shall be passed through at least one loop of copper tubing placed around the inside circumference of the cold bath so that the volume measurement is made at approximately 75 F. Precautions must be taken to avoid leaks at any one point in the system.

Note 3.—Delete this note.

New Note.—Add a new Note 5 to read as follows:

NOTE 5.—A wet test meter calibrated in hundredths of a liter is suitable.

Section 5.—After the first sentence of Paragraph (*a*), add: "The following test shall be carried out within 3 hr after heating the sample." After the present fourth sentence, add "Connect the air outlet tube to the air volume measuring device." In the present fifth sentence, change the time interval for air through the stone from "5 min ± 10 sec" to "5 min ± 3 sec." Change the last two sentences to read "The total air volume which has passed through the system shall be 470 ± 25 ml. Allow the cylinder to stand for 10 min ± 10 sec and again record the volume of foam."

Change Paragraph (*b*) to read as follows:

(*b*) Pour a second portion of sample into a cleaned 1000-ml cylinder until the liquid level is at the 180-ml mark. Immerse the cylinder at least to the 900-ml mark in the bath maintained at 200 ± 1 F. When the oil has reached a temperature of 199 ± 2 F, insert a clean diffuser stone and air-inlet tube and proceed as described in Paragraph (a), recording the volume of foam at the end of the blowing and settling periods.

New Section.—Add the following new Section 6, renumbering the subsequent sections accordingly:

6. *Alternative Procedure.*—For routine testing a simplified testing procedure may be used. This procedure differs from the standard method in only one respect. The total air volume used during the 5 min blowing period is not measured after the air has passed through the diffuser stone. This eliminates the volume measuring equipment and the air-tight connections necessary to carry the exit air from the graduated cylinder to the volume measuring device, but requires that the flowmeter be correctly calibrated and that the flow rate be carefully controlled.

Results obtained by this procedure shall be reported as D 892 - 58 T (Alternate).

New Section.—Add a new Section 7 to read as follows, renumbering the subsequent section accordingly:

7. *Precision.*—The following data should be used for judging the acceptability of results (95 per cent probability) according to the concept of precision as given in the ASTM Proposed Recommended Practice for Applying Precision Data given in ASTM Method of Test for Petroleum Products:

(a) Duplicate results by the same operator for foam volume at the end of the 5 min blowing period should not be considered suspect unless they differ by more than 10 ml or 15 per cent of their average value, whichever is larger.

(b) The result submitted by each of two laboratories for foam volume at end of 5 min blowing period should not be considered suspect unless they differ by more than 10 ml or 38 per cent of their average value, whichever is larger.

Appendix.—Add an Appendix as follows:

APPENDIX

TEST FOR MAXIMUM PORE DIAMETER AND PERMEABILITY OF STONE DIFFUSERS

(Based on a tentative method of test for rigid porous filters developed by Subcommittee 26 of ASTM Committee E-1 on Methods of Testing)

Definitions

1. (*a*) *Maximum Pore Diameter* is the diameter in microns of a capillary of circular cross-section which is equivalent (with respect to surface tension effects) to the largest pore in the diffuser under construction.

(*b*) *Permeability* is the flow of air, in milliliters per min, through the diffuser stone at air pressure of 250 mm of water.

Apparatus

2. (*a*) Apparatus for the maximum pore diameter determination consists of a regulated source of clean, dry compressed air; a U-tube water manometer of sufficient length to read a pressure differential of 800 mm; and a cylinder of a size sufficient (250 ml is suitable) to conveniently immerse a diffuser stone to a depth of 100 mm.

(*b*) Additional apparatus for permeability determination consists of a gas volume meter of sufficient capacity to measure flow rates of at least 6000 ml per min and a filtering flask large enough that 1-in. diameter diffuser stones will pass through the neck. The flask shall be fitted with a rubber stopper with a single hole to admit the air-inlet tube (see Fig. 4).

Method of Test

3. (*a*) *Maximum Pore Diameter*.—Support the clean diffuser by an air-inlet tube at a depth of 100 mm as measured to the top of the stone in distilled water in a cylinder and allow it to soak for at least 2 min. Connect the air-inlet tube to a controllable source of clean, compressed air and a manometer as shown in Fig. 3. Increase the air pressure at a rate of about 50 mm of water per min until the first dynamic bubble passes through the filter and rises through the water. The first dynamic bubble is recognized by being followed by a succession of additional bubbles. Read the water level in both legs of the manometer and record the difference as the pressure, p. The uniformity of distribution of pores approaching maximum pore size may be observed by gradually increasing the air pressure and noting the uniformity with which streams of bubbles are distributed over the surface.

Calculate maximum pore diameter, D, in microns from the following formula:

$$D = \frac{29{,}225}{p - 100}$$

(*b*) *Permeability*.—Connect the clean, dry diffuser stone with a controllable source of clean, dry compressed air and place it in a filtering flask connected to a suitable flowmeter as shown in Fig. 4. Adjust the pressure differential to 250 mm of water and measure the rate of flow of air through the diffuser stone in milliliters per minute. Depending on the sensitivity of the flowmeter used, this observation may be made for a suitably longer period and the average flow rate per minute recorded.

D 909 – 49 T,[7] Knock Characteristics of Aviation Fuels by the Supercharge Method:

Section 6 (*b*).—Change to read as follows:

> (*b*) **Compression Ratio,** 7.0 to 1 fixed by adjustment of the clearance volume to 108 ± 0.5 ml on cylinders of standard bore by the bench tilt procedure, Section 303 of Supplement III. Clearance volumes for oversize cylinders are shown in this section. If the detonation meter pickup is used the clearance volumes are 2 ml less than for the Waukesha plug.

Supplement I:

Section 104.—Change to read as recommended above for Method D 613.

Section 105 (*a*).—Change to read as recommended above for Method D 614.

Supplement III:

Section 303 (*c*).—Change to read as follows:

> (*c*) **Volume of Water, Supercharge.**—From a clean buret measure into the combustion chamber the volume of water specified in the following table:

Cylinder Size	Waukesha Plug, Solid or Thermal	Detonation Meter Pickup
Standard	108.0	106.0
0.010 in. Oversize	108.6	106.6
0.020 in. Oversize	109.2	107.2
0.030 in. Oversize	109.8	107.8

> Both the cylinder and water should be at the same temperature before starting the clearance volume measurement.

Section 410 (*b*).—Change as recommended above for Method D 613.

Table XVIII.—Change as recommended above for Method D 613.

D 974 – 55 T,[5] Test for Neutralization Value by Color-Indicator Titration:

Section 1 (*a*).—Change "benzene" to read "toluene."

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[7] 1952 ASTM Manual of Engine Test Methods for Rating Fuels.

Section 2.—Change "benzene" to read "toluene."

Section 6 (*c*).—Change to read as follows:

(*c*) *p-Naphtholbenzein Indicator Solution.*—The *p*-naphtholbenzein must meet the specifications given in the Appendix. Prepare a solution containing 10 g of *p*-naphtholbenzein[8] per liter of titration solvent (Paragraph (*e*)).

Footnote 8.—Change to read as follows:

[8] Solid *p*-naphtholbenzein to meet the specifications given in the Appendix is available from Distillation Products Industries, Eastman Organic Chemicals Department, Rochester 3, N. Y."

Section 6 (*e*).—Change "benzene" to read "toluene."

Section 12.—Change to read as follows:

12. *Precision.*—This precision section applies only to new, light-colored, straight mineral oils and new and used inhibited steam turbine oils. Insufficient data are available on other oils coming within the scope of this method so that no precision is given for such oils.

(*a*) *Repeatability.*—Duplicate determinations by the same operator should not differ by more than the following:

Neutralization Number	Repeatability
0.00 to 0.1	0.03
0.1 to 0.5	0.05
0.5 to 1.0	0.08
1.0 to 2.0	0.12

(*b*) *Reproducibility.*—Results from two different laboratories should not differ by more than the following:

Neutralization Number	Reproducibility
0.00 to 0.1	0.04
0.1 to 0.5	0.08
0.5 to 2.0	15 per cent of the neutralization number level

Note 9.—Delete the phrase "that are partially immiscible in the solvent or . . . ."

Note 10.—Delete this note.

Appendix.—Add the following appendix, which is to be published for only one year:

APPENDIX

SPECIFICATIONS FOR *p*-NAPHTHOLBENZEIN

Detailed Requirements:

p-Naphtholbenzein shall conform to the following requirements:

Appearance.—Red amorphous powder.

Chlorides.—Less than 0.5 per cent.

Solubility.—Ten grams shall dissolve completely in one liter of titration solvent (Section 5 (*c*)).

Minimum Absorbance.—Exactly 0.1000 g of sample is dissolved in 250 ml of methanol. Five ml of this solution is made up to 100 ml with pH 12 buffer. This final dilution should have a minimum absorbance of 1.20 when read at the 650 mμ peak using a Beckman DU Spectrophotometer, 1 cm cells and water as the blank.

pH Range.—(*a*) Indicator turns to the first clear green at a relative pH of 11 ± 0.5 when tested by the method for pHr range of p-naphtholbenzein indicator as described in Section 6 (*c*).

(*b*) Requires not more than 0.5 ml of 0.01 *N* KOH above that for blank to bring indicator solution to the first clear green.

(*c*) Requires not more than 1.0 ml of 0.01 *N* KOH above that for blank to bring indicator solution to a blue color.

(*d*) Initial pHr of indicator solution is at least as high as that of the blank.

Method for Determining pHr Range of p-Naphtholbenzein Indicator

Scope

1. This method is intended for determining the acceptability of *p*-naphtholbenzein indicator for use in ASTM Method D 974 with regard to color change over a pHr range.

Outline of Method

2. A prescribed amount of indicator is titrated electrometrically through the various color changes with alcoholic potassium hydroxide and results plotted against meter readings converted to pHr units.

Definitions

3. pHr. This is an arbitrary term which expresses the relative hydrogen ion activity in the toluene-isopropyl alcohol medium in a manner similar to that in which the term pH expresses

the actual hydrogen ion activity in aqueous solutions. For the purpose of this method, the pHr acidity scale is defined by two standard buffer solutions which have been designated pHr 4 and pHr 11. The exact relation between pHr and the true pH of a toluene-isopropyl alcohol solution is not known and cannot be readily determined.

Apparatus

4. *Meter, Glass Electrode, Calomel Electrode, Stirrer, Beaker and Stand*, as specified in the Appendix to ASTM Method D 664, Test for Neutralization Value (Acid and Base Numbers) of Petroleum Products (Potentiometric Titration Method).

Purity of Reagents

5. Reagent grade chemicals shall be used in all tests.

Unless otherwise indicated, it is intended that all reagents shall conform to the specifications established by the Committee on Analytical Reagents of the American Chemical Society where such specifications are available.[7] Other grades may be used, provided that it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

References to water shall be understood to mean distilled water.

Reagents

(*a*) *Standard Potassium Hydroxide Solution (Alcoholic, 0.2 N)*. Prepare, store and standardize as directed in ASTM Method D 664.

(*b*) *Standard Hydrochloric Acid Solution (Alcoholic, 0.2N)*.—Prepare and standardize as directed in ASTM Method D 664.

(*c*) *Titration Solvent*. Add 500 ml of toluene and 5 ml of water to 495 ml of anhydrous isopropyl alcohol. The titration solvent should be made up in large quantities, and should be tested as described in Section 8.

(*d*) *Acidic Buffer Solution, pHr = 4.0*.—Prepare a stock solution as directed in ASTM D 664. Add 10 ml of buffer stock solution to 100 ml of titration solvent. Use the diluted solution within one hour.

(*e*) *Alkaline Buffer Solution, pHr = 11.0*.—Prepare a stock solution as directed in ASTM D 664. Add 10 ml of buffer stock solution to 100 ml of titration solvent. Use the diluted solution within one hour.

(*f*) *Potassium Chloride Electrolyte*.—Prepare a saturated solution of potassium chloride in water.

(*g*) *p-Naphtholbenzein Indicator Solution*.—Prepare as in ASTM D 974.

Preparation of Electrode System

6. Prepare electrode system as in ASTM D 664, Section 7(*a*) and (*b*).

Standardization of Apparatus

7. (*a*) Prior to each test or series of tests, set the meter to read on the pH scale, insert the electrodes into a beaker containing the acidic nonaqueous buffer solution at a temperature of 25 ± 2 C and stir the solution vigorously. When the pH meter reading becomes constant adjust the asymmetry potential dial of the instrument so that the meter reads 4.0.

(*b*) Remove the acidic buffer, clean the electrodes, and immerse them in distilled water for several minutes. Dry the electrodes and insert them in a beaker containing alkaline nonaqueous buffer solution at 25 ± 2 C. When the pH meter reading has become steady, record the exact value. If the reading is within 0.2 pH units of 11.0, the initial acidity, pHr, of unknown solutions may be read directly from the dial of the meter. If the reading is not within 0.2 units of 11.0 prepare a correction graph as shown in Fig. 1. Use this graph to convert pH meter readings to initial acidity, pHr.

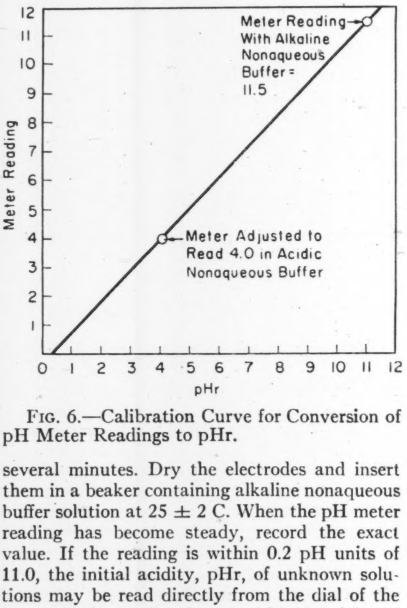

FIG. 6.—Calibration Curve for Conversion of pH Meter Readings to pHr.

Procedure

8. (*a*) Titrate 100 ml of titration solvent with 0.01 *N* KOH until the meter indicates a pHr between 13 and 14.

(*b*) Add 0.5 ml of indicator solution to a fresh portion of titration solvent and after cleaning the electrodes titrate with 0.01 *N* KOH until the meter indicates a pHr between 13 and 14.

(*c*) During the titration, plot the volume of titrant against the pHr or meter reading and note on the curve the various color changes at the corresponding pHr values.

NOTE 1.—The following color changes, in order, are intended as a guide:

Amber to olive green
Olive green to clear green
Clear green to bluish green
Bluish green to blue

(*d*) Plot the blank titration on the same paper used for the indicator.

Calculation

9. Subtract the volume of titrant used in the blank titration from that used for the indicator solution titration at the same pHr corresponding to the definite color changes between 10 and 12 pHr.

New Figure.—Add a new Fig. 1, as shown in the accompanying Fig. 6, to the new Appendix.

D 1019 - 57 T,[5] Test for Olefinic Plus Aromatic Hydrocarbons in Petroleum Distillates:

Section 6 (*a*).—Replace the second sentence with the following:

Place the flask in its holder in the shaking machine. Flasks held in a vertical position must be submerged at least to the 20 per cent (2 ml) mark in the ice water bath (0 to 2 C). Flasks held in the oblique position, that is, more than 10 deg from the vertical, must be immersed above the top graduation in an ice pack or ice water (Note 6). Keep the flask in the ice water or ice pack for at least 10 min before adding sample (Note 7).

New Note.—Add a new Note 6 to read as follows, renumbering the present Note 6 and subsequent notes accordingly.

NOTE 6.—One practical form of ice pack for the inclined flask is shown in Fig. 9.

D 1091 - 54 T,[3] Test for Phosphorus in Lubricating Oils, Additives and Concentrates:

Section 7 (*a*).—Change to read as follows:

(*a*) *Hydrogen Peroxide* (*30 per cent*), containing no more than 0.0002 per cent phosphorus.

D 1220 - 57 T,[8] Calibrating Upright Tanks:

Section 55 (*d*).—In Paragraph (*5*), change the Note to read as follows:

Note.—A total of —— bbl has been deducted from this table between —— ft —— in. and —— ft —— in. for roof displacement based upon a floating weight of — lb and an observed liquid gravity of ——° API as observed under conditions of liquid in which the roof is floating. (This may be at any observed temperature.) Gaged levels above —— ft —— in. reflect this deduction but should be corrected for actual observed gravity of the liquid at prevailing temperatures as follows: For ——° API observed, no correction. For each degree below ——° API observed —— Add —— bbl. For each degree above ——° API observed —— Substract —— bbl. ROOF DISPLACEMENT HAS BEEN DEDUCTED.

Section 52.—Change to read as follows:

52. (*a*) *Expansion and Contraction of Steel Tank Shells With Temperature.*—For non-insulated metal tanks, the temperature of the shell may, in general, be taken as the mean of the adjacent liquid and ambient temperatures on opposite sides at the same location. In which case:

$$t_S = \frac{t_L - t_A}{Z} \qquad (9)$$

(*b*) For insulated metal tanks, the temperature of the shell may be taken as closely approximating the adjacent liquid temperature. In which case, $t_S = t_L$.

Volume correction, per cent = **0.0013** ($t_S - 60$)

In applying these principles to upright cylindrical tanks, the horizontal cross-sectional area may be taken as a function of tank calibration. The coefficient 0.0013 is predicated on a thermal expansion coefficient 0.0000065 per degree Fahrenheit for low carbon steel. For metals other than low carbon steel, the coefficient 0.0013 should be corrected by dividing by the thermal expansion coefficient of low carbon steel and multiplying by the thermal expansion coefficient of the actual metal encountered. For example, if the tank shell is aluminum, the coefficient 0.0013 is changed to

$$0.0013 \times \frac{0.0000125}{0.0000065} = 0.0025$$

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[8] Issued as a separate reprint.

where:

0.0000125 = thermal expansion coefficient for aluminum, and

0.0000065 = thermal expansion coefficient for low carbon steel.

The liquid height dimension is a function of gaging the liquid level; accordingly, the effect of thermal expansion or contraction on innage and outage gage readings should be considered separately.

D 1319 - 56 T,[9] Test for Hydrocarbon Types in Liquid Petroleum Products (Fluorescent Indicator Adsorption (FIA) Method):

Section 1.—Change the last sentence to read "Samples containing appreciable amounts of C_5 and lighter hydrocarbons should be depentanized to avoid errors; the quantitative effect has not as yet been determined."

Section 4.—Add a new Paragraph (*d*) and new Note 5 to read as follows, relettering the present Paragraphs (*d*) and (*e*), and renumbering the present Note 5 and subsequent Notes accordingly:

(*d*) *Standard Dyed Gel*[7] consisting of a mixture of recrystallized Petrol Red AB4 and purified portions of the olefin and aromatic markers obtained by chromatographic adsorption following a definite, uniform procedure.

NOTE 5.—This mixture has been cooperatively tested and has been found to produce more brilliant and definite zones when applied to kerosine type fuels and is especially recommended for use in analyzing commercial turbine fuels for aircraft. Results from such use have given a precision somewhat better than stated in Section 7.

Footnote 7.—Change to include dyed gel.

Section 5 (a).—Add the following after the fifth sentence:

Rotate the column slowly by hand while vibrating or tapping to prevent horizontal segregation of particles and to avoid the possibility of channeling. Alternately vibrating racks have been used successfully for this purpose.

New Note.—Add the following new Note 10 after Section 5 (*f*), renumbering the subsequent note accordingly:

NOTE 10.—If the dye mixture specified in Section 4(*c*) or the dyed gel prepared as described in Note 4 are used, the use of a Wratten No. 47 or No. 50 light filter between the column and the eyes may aid in the recognition of the aromatic zone front.

D 1322 - 54 T,[5] Test for Smoke Point of Jet Fuels:

New Section.—Add new Section 5 to read as follows, renumbering the present Section 5 and subsequent sections accordingly:

5. *Reference Fuel Blends.*—The materials used to make the reference fuel blends are the ASTM Knock Test Reference Fuels, *Iso*-octane and Reference Fuel Grade Toluene. The composition and smoke points of these blends are as follows:

Toluene, per cent by volume	*Iso*octane, per cent by volume	Standard Smoke Point at 760 mm
40	60	14.7
25	75	20.2
15	85	25.8
10	90	30.2
5	95	35.0

The blends must be made very accurately. The use of either calibrated pipets or burets is recommended. Calibrated flasks should not be used.

Section 6.—Renumber as 8. At the end of Paragraph (*d*) (*3*) add "These are to be disregarded."

Section 7.—Renumber as 9. Change the second sentence of Paragraph (*a*) to read "Correct this average for both the barometric pressure by the use of Figs. 3 or 4 and the calibration factor in accordance with the procedure in Section 7."

In Paragraph (*b*), change "barometric pressure without any" to read "barometric pressure and the calibration curve without any."

New Section.—Add a new Section 7 to read as follows:

7. *Calibration of Apparatus.*—(*a*) The apparatus is calibrated by testing, in accordance with Section 8, two of the reference fuel blends in Section 5 which, if possible, bracket

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[9] 1956 Supplement to Book of ASTM Standards, Part 5.

the smoke point of the test fuel. If not possible, the two blends nearest the smoke point should be used. The determined smoke point of the test fuel is multiplied by a correction factor determined as follows:

$$\text{Correction Factor} = \frac{\frac{A_s}{A_d} + \frac{B_s}{B_d}}{2}$$

where:

A_s = standard smoke point for first reference fuel blend,

A_d = determined smoke point for first reference fuel blend,

B_s = standard smoke point for second reference fuel blend,

B_d = determined smoke point for second reference fuel blend.

If the determined smoke point of the test fuel exactly matches the determined smoke point of a reference fuel blend, the second bracketing reference fuel shall be the reference fuel blend with the next higher smoke point if there is one; otherwise the one with the next closest smoke point shall be used.

(*b*) The required reference fuel blends should be run each day by each operator and a test on a given reference fuel blend need not be repeated in any given day unless a change in the apparatus or operator is made.

Figure 2.—Make the notation "Base of Flame" at the bottom of the illustrated flame.

D 1368 – 55 T,[5] Test for Trace Concentrations of Tetraethyllead in Primary Reference Fuels:

Section 3.—Add the following new Paragraph (*a*), relettering the present Paragraph (*a*) and subsequent paragraphs accordingly:

(*a*) Chemically resistant glassware shall be used in this method.

D 1408 – 56 T,[7] Calibrating Spherical and Spheroidal Tanks:

Appendix III.—Change to read as follows:

PROCEDURE FOR COMPUTING THE VOLUME CORRECTION FOR THERMAL EXPANSION OR CONTRACTION OF SPHERICAL AND SMOOTH SPHEROIDAL TANKS WHICH ARE CONSTRUCTED OF METALS OTHER THAN LOW CARBON STEEL

The basis and method of correcting the volume of spherical and smooth spheroidal tanks which have been affected in size by changes in temperature is described in Section 10. The coefficient *K* is obtained from the curve of Fig. 7, which is based upon a thermal expansion coefficient, 0.0000065, for low carbon steel. The coefficient *K* varies linearly with the coefficient of thermal expansion. The value *K* for other tank metals may be derived by taking the value from the curve of Fig. 7, dividing by the thermal expansion coefficient for low-carbon steel, and multiplying by the thermal expansion coefficient of the actual metal encountered. For example:

$$K_A 1 = K_s \frac{(0.0000125)}{(0.0000065)}$$

where:

$K_A 1$ = volume correction coefficient for an aluminum shell,

K_s = volume correction coefficient for a low carbon steel shell (from Fig. 7),

0.0000125 = coefficient of thermal expansion for wrought aluminum, and

0.0000065 = coefficient of thermal expansion for low-carbon steel.

REVISION OF STANDARDS AND REVERSION TO TENTATIVES

The committee recommends revisions as indicated of the following standards and their reversion to tentative:

D 93 – 52,[5] Test for Flash Point by Means of the Pensky-Martens Closed Tester: Revise as appended hereto.[4]

D 1092 – 55,[5] Test for Apparent Viscosity of Lubricating Greases: Revise as appended hereto.[4]

ADOPTION OF TENTATIVES AS STANDARDS WITHOUT REVISION

The committee recommends that the following four tentative methods of test be approved for reference to letter ballot of the Society for adoption as standard without revision:

D 95 – 56 T,[9] Test for Water in Petroleum Products and Other Bituminous Materials

D 937 - 49 T,[5] Test for Penetration of Petrolatum

D 1269 - 56 T,[9] Test for Polarographic Determination of Tetraethyllead in Gasoline

D 1402 - 56 T,[9] Test for Effect of Copper on the Oxidation Rate of Grease

REVISIONS OF STANDARDS, IMMEDIATE ADOPTION

The committee recommends for immediate adoption revisions of the following six standards as indicated, and accordingly asks for the necessary nine-tenths affirmative vote at the Annual Meeting in order that the revisions may be referred to letter ballot of the Society:

D 129 - 57,[3] Test for Sulfur in Petroleum Products and Lubricants by the Bomb Method:

Section 7.—Change to read as follows:

7. *Precision*.—Results should not be considered suspect unless they differ by more than the following amounts (Note 5):

Sulfur, per cent by weight	Repeatability	Reproducibility
0 to 0.5........	0.04	0.05
0.5 to 1.0........	0.06	0.09
1.0 to 1.5........	0.08	0.15
1.5 to 2.0........	0.12	0.25
2.0 to 5.0........	0.18	0.27

NOTE 5.—The precision shown in the above table does not apply to samples containing over 2 per cent chlorine because an added restriction on the amount of sample which can be ignited is imposed.

D 189 - 52,[5] Test for Carbon Residue of Petroleum Products, Conradson Carbon Residue:

Section 4 (a).—Replace the last sentence with the following:

For samples showing more than 5.0 and less than 15.0 per cent carbon residue by the procedure described in Section 3(*a*), repeat the test using a 5 ± 0.5 g sample weighed to the nearest 5 mg. In the event that a result greater than 15.0 per cent is obtained, repeat the test, reducing the sample size to 3 ± 0.1 g. weighed to the nearest 5 mg.

If the sample boils over, reduce the sample size first to 5 g and then to 3 g as necessary to avoid the difficulty.

When the 3-g sample is used, it may not be possible to control the preignition and vapor burning times within the limits specified in Section 3(*c*), and in such cases, the results are nevertheless valid.

Section 5.—Change Paragraphs (*a*) and (*b*) to read as follows:

(*a*) *Repeatability*.—Duplicate results by the same operator should be considered suspect if they differ by more than the following amounts:

Conradson Carbon Residue, per cent	Repeatability, per cent of Mean
0.1 to 5.0............	see Fig. 2
5.1 to 20.0............	13

(*b*) *Reproducibility*.—The result submitted by one laboratory should not be considered suspect unless it differs from that of another laboratory by more than the following amounts:

Conradson Carbon Residue, per cent	Reproducibility, per cent of Mean
0.1 to 5.0................	See Fig. 3
5.1 to 20.0................	19

Figures 2 and 3.—Delete the portions beyond 5 average per cent residue.

Footnote 2.—Add the sentences "In 1958, additional data on precision were obtained, and the procedure was revised to include samples having high percentages of residue."

D 323 - 56,[9] Test for Vapor Pressure of Petroleum Products (Reid Method):

Section 4 (e).—In the last sentence change "pouring spout" to read "transfer connection."

Section 5 (c).—In the second sentence add "maintained at 100 ± 0.2 F" after "water bath."

Section 6.—In the first sentence of Paragraph (*a*), change "insert the chilled spout (see Fig. 2)." to read "insert the chilled transfer connection and air tube (see Fig. 2)." In the second sentence, change "pouring spout" to read "transfer connection."

Replace the first two sentences of Paragraph (*e*) with the following:

(*e*) *Preparation of Apparatus for Next Test.*—Disconnect the air chamber, gasoline chamber, and pressure gage (Note 5). Remove trapped fluid in the Bourdon tube by repeated centrifugal thrusts. This may be accomplished in the following manner. Hold the gage between the palms of the hands with the right hand on the face side and the threaded connection of the gage forward. Extend the arms forward and upward at an angle of 45 deg with the coupling of the gage pointing in the same direction. Swing the arms downward through an arc of about 135 deg so that the centrifugal force aids gravity in removing the trapped liquid. Repeat this operation three times to expel all liquid. Purge the pressure gage by directing a small jet of air into its Bourdon tube for at least 5 min.

New Note.—Add a new Note 5 to read as follows, renumbering the present Note 5 and subsequent notes accordingly:

NOTE 5.—In the case of crude oil, the Bourdon tube must be washed with a volatile solvent after each test.

Table I.—For the definitions of P_t and P_{100} add the word "absolute" after the words "square inch."

Section 16.—Change to read as follows:

16. *Precision.*—The following table shall be used as a basis for judging the acceptability of results (95 per cent probability):

(*a*) *Repeatabiliy.*—Duplicate results by the same operators should be considered suspect if they differ by more than the amounts specified.

Range	Repeatability (Same Operator and Apparatus)
0 to 5 lb	0.1
5 to 16 lb (except Avgas)	0.2
16 to 26 lb	0.3
Above 26 lb	0.4
Avgas (approximately 7 lb)	0.1

(*b*) *Reproducibility.*—The results submitted by each of two laboratories should not be considered suspect unless they differ by more than the amounts specified.

Range	Reproducibility (Different Operator and Apparatus)
0 to 5 lb	0.35
5 to 16 lb (except Avgas)	0.3
16 to 26 lb	0.4
Above 26 lb	0.7
Avgas (approximately 7 lb)	0.15

D 357 - 56,[10] Test for Knock Characteristics of Motor Fuels by the Motor Method:

Section 6 (*n*).—Change to read as follows:

(*n*) *Micrometer Adjustment.*—Set to read 0.500 in. (5.5 to 1 compression ratio) with the piston at tdc and with a clearance volume of 140 ± 0.5 ml to the top face of the pick-up hole on cylinders of standard bore as measured by either of the tilt procedures described in Section 302 and 303 of Appendix III. Clearance volumes for oversize cylinders are shown in Section 302.

In addition, the following changes are to be made in the supplementary material to Method D 357 which appears in the 1956 ASTM Manual for Rating Motor Fuels by Motor and Research.

Supplement I:

Section 104.—Change to read as recommended above for Method D 613.

Section 105.—Add the following sentence to the first paragraph: "Cylinders of standard bore are preferred equipment but rebored cylinders up to a maximum of 0.030 in. oversize may be used."

Supplement IV:

Section 410 (*b*).—Change to read as recommended above for Method D 613.

Table XI.—Change to read as recommended above for Method D 613.

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[10] 1957 Supplement to ASTM Manual for Rating Motor Fuels by Motor and Research Methods.

D 664 – 54,[5] Test for Neutralization Value (Acid and Base Numbers) of Petroleum Products (Potentiometric Titration):

Section 3.—Change "benzene" to read "toluene."

Section 6 (j).—Change "benzene" to read "toluene."

Note 8.—Change "benzene" to read "toluene."

D 908 – 56,[10] Test for Knock Characteristics of Motor Fuels by the Research Method:

Section 6 (m).—Change to read as recommended above for Section 6(*n*) of Method D 357.

Sections 104, 105, 410 (b) and Table XI.—Change to read as indicated above for Method D 357.

REVERSION OF STANDARD TO TENTATIVE WITHOUT REVISION

The committee recommends the reversion to tentative of the Standard Method of Test for Autogenous Ignition Temperatures of Petroleum Products (**D 286 – 30**)[5] in order to facilitate a major revision that is under consideration.

TENTATIVES CONTINUED WITHOUT REVISION

The committee has reviewed the following 17 tentative methods of test that have stood two or more years in their present form, and for good and sufficient reason discussed within the committee, recommends that they be continued as tentatives without revision:

D 94 – 56 T, Test for Saponification Number of Petroleum Products by Color Indicator Titration
D 155 – 45 T, Test for Color of Lubricating Oil and Petrolatum by Means of ASTM Union Colorimeter
D 156 – 53 T, Test for Saybolt Color of Refined Petroleum Products (Saybolt Chromometer Method)
D 217 – 52 T, Test for Cone Penetration of Lubricating Grease
D 270 – 55 T, Method of Sampling Petroleum and Petroleum Products
D 396 – 48 T, Specifications for Fuel Oils
D 893 – 52 T, Test for Normal Pentane and Benzene Insolubles in Used Lubricating Oils
D 1086 – 56 T, Method of Measuring the Temperature of Petroleum and Petroleum Products
D 1218 – 52 T, Methods of Measurement of Refractive Index and Refractive Dispersion of Hydrocarbon Liquids
D 1318 – 54 T, Test for Sodium in Residual Fuel Oil by Flame Photometer
D 1367 – 55 T, Test for Lubricating Qualities of Graphites
D 1401 – 56 T, Test for Emulsion Characteristics of Steam-Turbine Oils
D 1403 – 56 T, Test for Cone Penetration of Lubricating Grease Using One-Quarter Scale Cone Equipment (One-Quarter Scale of Method D 217 Cone and Worker)
D 1404 – 56 T, Test for Estimation of Deleterious Particles in Lubricating Grease
D 1406 – 56 T, Methods for Liquid Calibration of Tanks
D 1407 – 56 T, Methods for Calibrating Barge Tanks
D 1410 – 56 T, Methods for Calibrating Stationary, Horizontal Tanks

STANDARDS CONTINUED WITHOUT REVISION

The committee has reviewed the following 21 standards which have stood for six years or more without revision and which in the opinion of the committee represent good practice. It recommends that they be continued as standards without revision:

D 91 – 52, Test for Precipitation Number of Lubricating Oils
D 127 – 49, Test for Melting Point of Petrolatum and Microcrystalline Wax
D 187 – 49, Test for Burning Quality of Kerosine
D 219 – 36, Test for Burning Quality of Long-Time Burning Oil for Railway Use
D 239 – 30, Test for Burning Quality of Mineral Seal Oil
D 341 – 43, Standard Viscosity-Temperature Charts for Liquid Petroleum Products
D 473 – 48, Test for Sediment in Fuel Oil by Extraction
D 482 – 46, Test for Ash Content of Petroleum Oils
D 566 – 42, Test for Dropping Point of Lubricating Grease
D 612 – 45, Test for Carbonizable Substances in Paraffin Wax

- **D 810 - 48,** Test for Sulfated Residue, Lead, Iron, and Copper in New and Used Lubricating Oils
- **D 811 - 48,** Methods of Chemical Analysis for Metals in Lubricating Oils
- **D 942 - 50,** Test for Oxidation Stability of Lubricating Greases by the Oxygen Bomb Method
- **D 971 - 50,** Test for Interfacial Tension of Oil Against Water by the Ring Method
- **D 973 - 50,** Test for Butadiene Content of Polymerization Grade Butadiene
- **D 1017 - 51,** Test for Benzene and Toluene by Ultraviolet Spectrophotometry
- **D 1020 - 52,** Test for Acetylene in Polymerization-Grade Butadiene (Silver Nitrate Method)
- **D 1023 - 52,** Test for Separation of Residue from Butadiene
- **D 1025 - 52,** Test for Nonvolatile Residue of Polymerization Grade Butadiene
- **D 1026 - 51,** Test for Sodium in Lubricating Oils and Lubricating Oil Additives
- **D 1093 - 52,** Test for Acidity of Residue from Distillation of Gasoline and of Petroleum Solvents

AMERICAN STANDARDS

The committee recommends the following for action by ASA Sectional Committee Z11 on Petroleum Products and Lubricants:

Approval as American Standard:

Test for Polarographic Determination of Tetraethyllead in Gasoline (D 1269 - 56 T),
Test for Effect of Copper on the Oxidation Rate of Grease (D 1402 - 56 T),

Reaffirmation as American Standard:

Z11.51—1943, Test for Dropping Point of Grease (D 566 - 42),

Approval as American Standard as Revised in This Report:

Z11.7, Flash Point by Pensky-Martens Closed Tester (D 93),
Z11.25, Carbon Residue of Petroleum Products (D 189),
Z11.36, Existent Gum in Fuels (D 381),
Z11.37, Knock Characteristics of Motor Fuels by the Motor Method (D 357),
Z11.44, Vapor Pressure of Petroleum Products (D 323),
Z11.47, Carbon Residue of Petroleum Products, Ramsbottom Coking Method (D 524),
Z11.59, Neutralization Value by Potentiometric Titration (D 664),
Z11.69, Knock Characteristics of Motor Fuels by the Research Method (D 908),
Z11.71, Olefinic Plus Aromatic Hydrocarbons in Petroleum Distillates (D 1019),
Z11.72, Apparent Viscosity of Lubricating Grease (D 1092),
Z11.78, Foaming Characteristics of Crankcase Oils (D 892).

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[11]

This report has been submitted to letter ballot of the committee, which consists of 138 voting members; 97 members have returned their ballots, of whom 90 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

H. M. SMITH,
Chairman.

W. T. GUNN,
Secretary.

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[11] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee D-2 presented to the Society through the Administrative Committee on Standards the following recommendations:

Revision of Tentative Specification for:

Gasoline (D 439 - 58 T).

Revision of Tentative Method for:

Color of Petroleum Products (D 1500 - 57 T).

Withdrawal of Tentative Methods for:

Calibrating Tank Car Tanks (Pressure and Non-Pressure Types) (D 1409 - 56 T).

These recommendations were accepted by the Standards Committee on September 9, 1958, and the revised tentative specification and method appear in the 1958 Book of ASTM Standards, Part 7.

REPORT OF COMMITTEE D-3

ON

GASEOUS FUELS*

Since the committee submitted its last report, resignations of the following members have occurred: L. T. Bissey, chairman; R. V. Warrick, secretary; J. F. Anthes, Advisory Committee; H. S. Bean, chairman, Subcommittee II on Measurement of Gaseous Samples; and T. S. Bacon, chairman, Subcommittee IV on Determination of Specific Gravity and Density of Gaseous Fuels.

E. E. Stovall has been elected to succeed Mr. Bacon as chairman of Subcommittee IV. K. R. Knapp has been reappointed by the Advisory Committee as secretary.

The officers and members of the Advisory Committee elected for the ensuing term of two years are as follows:

Chairman, D. V. Kniebes.

Vice-Chairman, F. E. Vandaveer.

Secretary, K. R. Knapp.

Advisory Committee: D. V. Kniebes, Chairman; and W. M. Cline, A. W. Gauger, E. G. Hammerschmidt, K. R. Knapp, G. C. Putnam, and F. E. Vandaveer.

REVISION OF STANDARD, IMMEDIATE ADOPTION

The committee recommends for immediate adoption revisions as follows of the Standard Method of Test for Water Vapor Content of Gaseous Fuels by Measurement of Dew-Point Temperature (**D 1142 – 53**),[1] and accordingly asks for the necessary nine-tenths affirmative vote at the Annual Meeting in order that the revisions may be referred to letter ballot of the Society. The formula shown, together with accompanying tables and figure, provides a ready and convenient means for determining water vapor content under usual conditions.

New Paragraph.—To Section 5 add a new Paragraph (*c*) to read as follows:

(*c*) A correlation of the available data on the equilibrium water content of natural gases has been reported by Bukacek.[7] This correlation is believed to be accurate enough for the requirements of the gaseous fuels industry except for unusual situations where the dew point is measured at conditions close to the critical temperature of the gas. The correlation is a modified form of Raoult's law having the following form:

$$W = A/P + B$$

where:

W = water vapor content, lb per million SCF,
P = total pressure, psia,
A = a constant proportional to the vapor pressure of water, and
B = a constant depending on temperature and gas composition.

Values of B were computed from available data on methane, methane-ethane mixtures, and natural gases.

Table II lists values of the constants A and B for natural gases in the temperature range of −40 to 460 F. Table III lists values of water vapor content from −40 to 250 F and from 14.7 psi to 5000 psi, covering the range of most natural gas processing applications.

A convenient graphical representation of the data in Table III is illustrated in Fig. 2. The moisture content values given can be corrected to base conditions other than 14.7 psia and 60 F by the same equations given in Table II. Complete sets of these charts covering the entire range of pressures and temperatures of Table III may be purchased from the Institute of Gas Technology.

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* Sixty-first Annual Meeting of the Society, June 22–27, 1958.

[1] 1955 Book of ASTM Standards, Part 5.

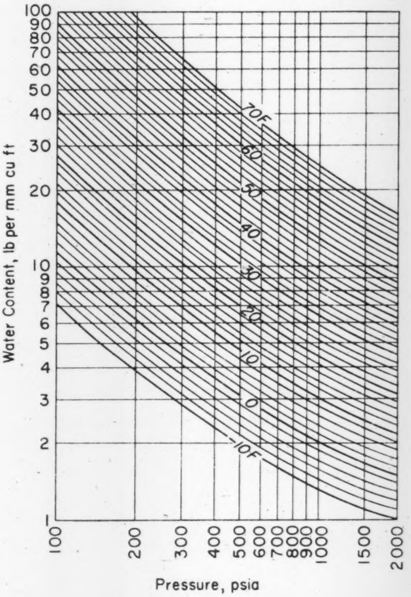

FIG. 1.—Saturation Water Content of Natural Gases.

New Tables.—Add new Tables II and III as shown in the accompanying Tables I and II.[2]

New Figure.—Add a new Fig. 2 as shown in the accompanying Fig. 1.

REAPPROVAL OF STANDARD

The committee recommends the reapproval of the Standard Method of Test for Specific Gravity of Gaseous Fuels **(D 1070 – 52)**[1] which has stood for six years without revision.

TENTATIVE CONTINUED WITHOUT REVISION

The committee recommends the continuation without revision of the Tentative Method of Analysis of Carbureted Water Gas by the Mass Spectrometer **(D 1302 – 53 T)**.[1]

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[3]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee IV on Determination of Specific Gravity and Density of Gaseous Fuels (E. E. Stovall, chairman) recommended the reapproval of Methods D 1070 referred to earlier in the report.

Subcommittee VI on Determination of Water Vapor Content of Gaseous Fuels (E. G. Hammerschmidt, chairman) reviewed the results of extensive work by the Institute of Gas Technology on a method for determining equilibrium water content of natural gases. This enables determinations to be made by the use of a modified form of Raoult's law with accompanying tables and charts. The method is believed sufficiently accurate for the gaseous fuels industry except for unusual conditions where dew-point measurement is made at conditions close to the gas critical temperature. By letter ballot of the subcommittee its inclusion in Method D 1142 was, therefore, recommended through addition of Paragraph (*c*) to Section 5, Calculations.

Subcommittee VII on Complete Analysis of Chemical Composition of Gaseous Fuels (D. V. Kniebes, chairman) recommended the continuation as tentative of

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[7] R. F. Bukacek, "Equilibrium Moisture Content of Natural Gases," Institute of Gas Technology Research Bulletin 8 (1955). Reports work sponsored by the Pipeline Research Committee of the American Gas Association.

[2] The new tables are not included in this report but appear in the revised Method of Test, see 1958 Book of ASTM Standards, Part 8.

[3] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

Method D 1302 – 53 T. This action was taken in view of contemplated revisions of this standard to meet some objections of Committee E-14 on Mass Spectrometry to acceptance in its present form.

This report has been submitted to letter ballot of the committee, which consists of 34 members; 26 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

F. E. Vandaveer,
Vice-Chairman.

K. R. Knapp,
Secretary.

REPORT OF COMMITTEE D-4

ON

ROAD AND PAVING MATERIALS*

Committee D-4 on Road and Paving Materials held two meetings during the year: in Atlantic City, N. J., on June 21, 1957, and in St. Louis, Mo., on February 14, 1958.

The committee has unanimously elected Julius Adler and R. R. Thurston as Honorary Members of the committee.

The committee has discharged Subcommittee B-25 on Effect of Water on Compressed Bituminous Mixtures and Subcommittee D-4 on Asphalt Planks. The title of Subcommittee D-3 has been changed to Joint Fillers and Asphalt Planks.

The committee is planning a symposium for the 1958 Annual Meeting on "Effect of Water on Bituminous Paving Mixtures." It is also planning a symposium for the 1959 Annual Meeting on "The Practical and Statistical Significance of Tests and Properties of Bituminous Paving Binders."

Plans are being made to sponsor a committee session during the Third Pacific Area National Meeting to be held in San Francisco, Calif., on October 11 to 16, 1959.

A few subcommittees now have a rather large membership and consideration is being given by the committee to placing some limitation on subcommittee membership.

The committee has jurisdiction over a number of ASTM Standards jointly with other committees and a study is being made to determine if such joint jurisdiction can be minimized.

The officers elected for the ensuing term of two years are as follows:

Chairman, A. B. Cornthwaite.
First Vice-Chairman, R. E. Bollen.
Second Vice-Chairman, J. E. Gray.
General Secretary, J. M. Griffith.
Membership Secretary, D. W. Lewis.

NEW TENTATIVES

The committee recommends for publication as tentative the following methods as appended hereto:[1]

Tentative Method of Test for:

- Density of Soil in Place by the Sand-Cone Method,
- Moisture-Density Relations of Soils Using a 10-lb Rammer and an 18-in. Drop,
- Resistance to Plastic Flow of Bituminous Mixtures by Means of the Marshall Apparatus,
- Resistance to Deformation and Cohesion of Bituminous Mixtures by Means of Hveem Apparatus,
- Moisture-Penetration Resistance Relations of Fine-Grained Soils.

Tentative Method of:

- Preparing Bituminous Mixtures by Means of California Kneading Compactor.

REVISIONS OF TENTATIVES

The committee recommends revisions as indicated of the following tentatives and their continuation as tentative:

Tentative Method of Test for Moisture-Density Relations of Soils (D 698 - 57 T):[2]

Title.—Change the title to read: "Tentative Methods of Test for Moisture-Density Relations of Soils, Using 5.5-lb Rammer and a 12-in. Drop."

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* Sixty-first Annual Meeting of the Society, June 22–27, 1958.

[1] The new and revised tentatives and standards appear in the 1958 Book of ASTM Standards, Part 4.

[2] 1957 Supplement to Book of ASTM Standards, Part 3.

Section 2(i).—Delete this paragraph.

Section 4.—In Paragraph (*a*) delete all but the first sentence. Change the last part of the Note at the end of Section 4, beginning with line 9, to read as follows:

> In these cases, the separate samples shall be thoroughly mixed with amounts of water sufficient to cause the moisture contents of the samples to vary by approximately 2 percentage points. The moisture contents selected shall bracket the optimum moisture content, thus providing samples which, when compacted, will increase in weight to the maximum density and then decrease in weight. The soil-water mixtures should be placed in covered containers and allowed to stand for not less than 12 hr before making the moisture-density test.

Section 7(b).—Change to read as follows:

> (*b*) Sieve an adequate quantity of the representative pulverized soil over the ¾-in. sieve. Discard the coarse material, if any, retained on the ¾-in. sieve (Note).
>
> NOTE.—If it is advisable to maintain the same percentage of coarse material (passing a 2-in. sieve and retained on a No. 4 sieve) in the moisture-density sample as in the original field sample, the material retained on the ¾-in. sieve shall be replaced as follows: Sieve an adequate quantity of the representative pulverized soil over the 2-in. and ¾-in. sieves. Discard the coarse material retained on the 2-in. sieve. Remove the material passing the 2-in. sieve and retained on the ¾-in. sieve and replace it with an equal weight of material passing the ¾-in. sieve and retained on the No. 4 sieve. Take the material for replacement from the remaining portion of the sample.

Section 8.—In Paragraph (*a*) delete all but the first sentence. In Paragraph (*b*) replace the last two sentences with the following:

> Following compaction, remove the extension collar, carefully trim the compacted soil even with the top of the mold by means of a straightedge. Holes developed in the surface by the removal of coarse material shall be patched with smaller size material. Weigh the mold and moist soil. Multiply the weight of the compacted specimen and mold, minus the weight of the mold, by 30, and record the result as the wet weight per cubic foot of the compacted soil.

Section 13.—Add a fourth item to read: "(*4*) In Methods C and D indication of removal or replacement of ¾-in. material."

Tentative Method of Test for Compressive Strength of Bituminous Mixtures (D 1074 – 55 T):[3] Revise as appended hereto.[1]

TENTATIVE REVISIONS OF STANDARDS

The committee recommends tentative revisions as indicated of the following standards:

Standard Methods of Testing Emulsified Asphalts (D 244 – 55):[3]

Section 8.—Replace the present Section 8 with a new section entitled "Precision," to read as follows:

> 8. The following data should be used for judging the acceptability of results (95 per cent probability):
>
> (*a*) Duplicate results by the same operator should be considered suspect if they differ by more than the following amount:

Water Content, per cent by Weight	Repeatability, per cent by Weight	
30 to 50	0.8	Item A

> (*b*) The results submitted by each of two laboratories should not be considered suspect unless they differ by more than the following amount:

Water Content, per cent by Weight	Reproducibility, per cent by Weight	
30 to 50	2.0	Item B

New Section.—Add a new Section 11 entitled "Precision" after the present Section 10 to read as follows, renumbering subsequent sections accordingly:

> 11. *Precision.*—The following data should be used for judging the acceptability of results (95 per cent probability):
>
> (*a*) Duplicate results by the same operator should be considered suspect if they differ by more than the following amount:

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[3] 1955 Book of ASTM Standards, Part 3.

Residue by Distillation, per cent by Weight	Repeatability, per cent by Weight	
50 to 70	1.0	Item A

(*b*) The results submitted by each of two laboratories should not be considered suspect unless they differ by more than the following amount:

Residue by Distillation, per cent by Weight	Reproducibility, per cent by Weight	
50 to 70	2.0	Item B

New Section.—Add a new Section 15 entitled "Precision" after the present Section 13, to read as follows:

15. *Precision.*—The following data should be used for judging the acceptability of results (95 per cent probability):

(*a*) Duplicate results by the same operator should be considered suspect if they differ by more than the following amount:

Residue by Evaporation, per cent by Weight	Repeatability, per cent by Weight	
50 to 70	0.4	Item A

(*b*) The results submitted by each of two laboratories should not be considered suspect unless they differ by more than the following amount:

Residue by Evaporation, per cent by Weight	Reproducibility, per cent by Weight	
50 to 70	0.8	Item B

New Section.—Replace the present Section 16 with a new Section 18, entitled "Precision," to read as follows:

18. *Precision.*—The following data should be used for judging the acceptability of results (95 per cent probability):

(*a*) Duplicate results by the same operator should be considered suspect if they differ by more than the following amount:

Test Temperature, deg Fahr	Viscosity, sec	Repeatability, per cent of the Mean	
77	20 to 100	5	Item A
122	75 to 400	10	

(*b*) The results submitted by each of two laboratories should not be considered suspect unless they differ by more than the following amount:

Test Temperature, deg Fahr	Viscosity, sec	Reproducibility, per cent of the Mean	
77	20 to 100	15	Item B
122	75 to 400	40	

New Section.—Add a new Section 22 entitled "Precision" after the present Section 19, to read as follows:

22. *Precision.*—The following data should be used for judging the acceptability of results (95 per cent probability):

(*a*) Duplicate results by the same operator should be considered suspect if they differ by more than the following amount:

Demulsibility, per cent by Weight	Repeatability, per cent of the Mean	
30 to 100	10	Item A

(*b*) The results submitted by each of two laboratories should not be considered suspect unless they differ by more than the following amount:

Demulsibility, per cent by Weight	Reproducibility, per cent of the Mean	
30 to 100	50	Item B

New Section.—Add a new Section 25 entitled "Precision" after the present Section 21, to read as follows:

25. *Precision.*—The following data should be used for judging the acceptability of results (95 per cent probability):

(*a*) Duplicate results by the same operator should be considered suspect if they differ by more than the following amount:

Settlement, per cent by Weight	Repeatability	
0 to 1.0	0.4 per cent by weight	Item A
above 1.0	5 per cent of the mean	

(*b*) The results submitted by each of two laboratories should not be considered suspect unless they differ by more than the following amount:

Settlement, per cent by Weight	Reproducibility	
0 to 1.0	0.8 per cent by weight	Item B
above 1.0	10 per cent of the mean	

New Section.—Add a new Section 30 entitled "Precision" after the present Section 25, to read as follows:

30. *Precision.*—The following data should be used for judging the acceptability of results (95 per cent probability):

(*a*) Duplicate results by the same operator should be considered suspect if they differ by more than the following amount:

Cement Mixing, per cent by Weight	Repeatability, per cent by Weight	
0 to 2	0.2	Item A

(*b*) The results submitted by each of two laboratories should not be considered suspect unless they differ by more than the following amount:

Cement Mixing, per cent by Weight	Reproducibility, per cent by Weight	
0 to 2	0.4	Item B

New Section.—Add a new Section 34 entitled "Precision" after the present Section 28, to read as follows:

34. *Precision.*—The following data should be used for judging the acceptability of results (95 per cent probability):

(*a*) Duplicate results by the same operator should be considered suspect if they differ by more than the following amount:

Sieve Test, per cent by Weight	Repeatability, per cent by Weight	
0 to 0.1	0.03	Item A

(*b*) The results submitted by each of two laboratories should not be considered suspect unless they differ by more than the following amount:

Sieve Test, per cent by Weight	Reproducibility, per cent by Weight	
0 to 0.1	0.08	Item B

New Section.—Add a new Section 47 entitled "Precision" after the present Section 40, to read as follows:

47. *Precision.*—The following data should be used for judging the acceptability of results (95 per cent probability):

(*a*) Duplicate results by the same operator should be considered suspect if they differ by more than the following amount:

Penetration of Residue, Range	Repeatability, Points	
80 to 200	15	Item A

(*b*) The results submitted by each of two laboratories should not be considered suspect unless they differ by more than the following amount:

Penetration of Residue, Range	Reproducibility, Points	
80 to 200	30	Item B

Standard Specifications for Hot-Mixed, Hot-Laid Asphaltic Concrete for Base and Surface Courses (D 947 – 55):[3]

Table II.—Revise the column headed "Grading No. 2" to read as follows:

Sieve Size	Grading No. 2
No. 4	100
No. 8	95 to 100
No. 16	85 to 100
No. 30	65 to 90
No. 50	30 to 60
No. 100	5 to 25
No. 200	0 to 5

Table III.—Revise the amounts passing the No. 200 sieve as follows:

Nominal Maximum Size of Aggregate	Amount Finer than No. 200 Sieve
2 in.	0 to 4
1½ in.	0 to 5
1 in.	1 to 7
¾ in.	2 to 8
½ in.	2 to 9
⅜ in.	2 to 10

Standard Specifications for Asphaltic Mixtures for Sheet Asphalt Pavements (D 978 – 54):[3]

Table II.—Revise the column headed "Grading No. 2" to read as follows:

Sieve Size	Grading No. 2
No. 4	100
No. 8	95 to 100
No. 16	85 to 100
No. 30	65 to 90
No. 50	30 to 60
No. 100	5 to 25
No. 200	0 to 5

Standard Specifications for Bituminous Mixing Plant Requirements (D 995 – 55):[3]

Section 2(e).—To the end of the paragraph add the sentence "The screens provided for separation of the aggregate into bins shall have an efficiency of not less than 85 per cent, based on laboratory sieves."

Standard Specifications for Fine Aggregate for Sheet Asphalt and Bituminous Concrete Pavements (D 1073 – 54):[3]

Section 3(a).—In the table of gradings revise the column headed "Grading No. 2" to read as follows:

Sieve Size	Grading No. 2
No. 4	100
No. 8	95 to 100
No. 16	85 to 100
No. 30	65 to 90
No. 50	30 to 60
No. 100	5 to 25
No. 200	0 to 5

REVISION OF STANDARD AND REVERSION TO TENTATIVE

The committee recommends revisions as appended hereto[1] of Standard Methods of Sampling Stone, Slag, Gravel, Sand, and Stone Block for Use as Highway Materials (**D 75 – 48**)[3] and reversion of the method to tentative.

ADOPTION OF TENTATIVES AS STANDARD WITHOUT REVISION

The committee recommends that the following tentatives be approved for reference to letter ballot of the Society for adoption as standard without revision:

Tentative Method of Test for:

Water in Petroleum Products and Other Bituminous Materials (**D 95 – 56 T**).[4]

Recommended Practice for:

Quantities of Materials for Bituminous Surface Treatments (**D 1369 – 55 T**).[3]

REVISIONS OF STANDARDS, IMMEDIATE ADOPTION

The committee recommends for immediate adoption revisions as indicated of the following standards, and accordingly asks for the necessary nine-tenths affirmative vote at the Annual Meeting in order that the revisions may be referred to letter ballot of the Society:

Standard Method of Test for Bitumen Content of Paving Mixtures by Centrifuge (D 1097 – 57):[3]

Section 5(d).—Delete the last three sentences and replace with the following:

> Ash the bituminous material at a dull red heat (500 to 600 C), cool and add 5 ml of saturated ammonium carbonate solution per gram of ash. Digest at room temperature for 1 hr and then dry in an oven at 110 C to constant weight, cool in a desiccator and weigh. Calculate the weight of ash in the entire volume of extract.

Standard Specifications for Sodium Chloride (D 632 – 43):[3] Revise as appended hereto.[1]

WITHDRAWAL OF TENTATIVE

The committee recommends the withdrawal of the Tentative Specifications for Preformed Expansion Joint Fillers for Concrete (Nonextruding and Resilient Types) (**D 544 – 56 T**).[4]

REAPPROVAL OF STANDARDS

The committee recommends the reapproval of the following standards which have stood for six or more years without revision:

Standard Method of Test for:

Abrasion of Rock by Use of the Deval Machine (**D 2 – 33**),
Toughness of Rock (**D 3 – 18**),
Bitumen (**D 4 – 52**),
Penetration of Bituminous Materials (**D 5 – 52**),
Softening Point of Bituminous Materials (Ring-and-Ball Method) (**D 36 – 26**),

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[4] 1956 Supplement to Book of ASTM Standards, Part 3.

Softening Point of Tar Products (Cube-in-Water Method) (**D 61 – 38**),
Specific Gravity of Road Oils, Road Tars, Asphalt Cements, and Soft Tar Pitches (**D 70 – 52**),
Specific Gravity of Asphalts and Tar Pitches Sufficiently Solid to be Handled in Fragments (**D 71 – 52**),
Ductility of Bituminous Materials (**D 113 – 44**),
Proportion of Bitumen Soluble in Carbon Tetrachloride (**D 165 – 42**),
Hot Extraction of Asphaltic Materials and Recovery of Bitumen by the Modified Abson Procedure (**D 762 – 49**),
Sulfonation Index of Road Tars (**D 872 – 48**),
Resistance to Plastic Flow of Fine-Aggregate Bituminous Mixtures (**D 1138 – 52**),
Amount of Material Finer Than No. 200 Sieve in Aggregate (**C 117 – 49**),
Specific Gravity and Absorption of Coarse Aggregate (**C 127 – 42**), and
Sieve Analysis of Fine and Coarse Aggregates (**C 136 – 46**).

Standard Specifications for:

Recut Granite Block for Pavements (**D 131 – 39**),
Asphalt Filler for Brick Pavements (**D 241 – 43**),
Tar (**D 490 – 47**),
Asphalt Plank (**D 517 – 50**),
Cut-Back Asphalt (Rapid Curing Type) (**D 597 – 46**), and
Cut-Back Asphalt (Medium Curing Type) (**D 598 – 46**).

Standard Methods of:

Float Test for Bituminous Materials (**D 139 – 49**), and
Sampling Bituminous Paving Mixtures (**D 979 – 51**).

Standard Recommended Practice for:

Bituminous Mixing Plant Inspection (**D 290 – 51**).

Standard Volume Correction Table for:

Tar and Coal-Tar Pitch (**D 633 – 44**).

EDITORIAL CHANGES

The committee recommends editorial changes as indicated in the following standards.

Standard Method of Test for Resistance to Plastic Flow of Fine-Aggregate Mixtures (D 1138 – 52):[3]

Title.—Change the title to read "Method of Test for Resistance to Plastic Flow of Fine-Aggregate Mixtures Using Hubbard-Field Apparatus."

Tentative Method of Test for Moisture or Volatile Distillates in Bituminous Mixtures (D 1461 – 57 T):[2]

Title.—Change the title to read "Tentative Method of Test for Moisture or Volatile Distillates in Bituminous Paving Mixtures."

Section 1.—In line 4 insert the word "paving" before "mixtures."

Section 2.—Change Paragraph (*c*) (*1*) to read as follows:

(*1*) For determination of water in bituminous paving mixtures, a glass trap of 10- or 25-ml capacity shall be used. The traps shall be graduated in 0.1-ml divisions with a ±0.05-ml maximum error below 1 ml, and in 0.2-ml divisions with a ±0.1-ml maximum error above 1 ml, as specified in ASTM Specifications E 123.

Change Paragraph (*d*) to read as follows:

(*d*) *Solvent.*—For general use an aromatic solvent is preferred since it has high solvency and dispersing power for most bituminous materials. Xylol, or a blend of 20 per cent benzol and 80 per cent xylol, is recommended. For asphalts and similar petroleum products, a petroleum distillate, 5 per cent boiling between 194 and 212 F, and 90 per cent distilling below 410 F, may be used. For coal-tar, water-gas tar, and similar materials, the aromatic solvent must be used.

Section 4.—Change the last sentence to read as follows: "Thoroughly break up this sample to avoid large lumps and place it in the still."

Standard Methods of Test for Specific Gravity and Absorption of Fine Aggregate (C 128 – 57):[3]

Section 3.—Add at the end of the section the sentence, "Mechanical aids, if desired, may be employed to assist in achieving the specified saturated surface-dry condition."

The recommendations in this report have been submitted to letter ballot of

the committee, the results of which will be reported at the Annual Meeting.[5]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee A-4 on Papers (W. H. Goetz, chairman) has been active in the solicitation of suitable technical papers for presentation at meetings of the Society. Several such papers have been offered for presentation at the 1958 Annual Meeting.

Subcommittee B-1 on Asphalt Content (H. F. Waller, Jr., chairman) has been engaged in detailed studies of various aspects of the test procedure outlined in the Tentative Method of Test for Vacuum Distillation of Liquid and Semi-Solid Asphaltic Materials to Obtain a Residue of Specified Penetration (D 1189 - 52 T). It has recommended that this method be retained as tentative in view of probable modifications resulting from studies now under way.

Subcommittee B-2 on Physical Tests for Compressed Bituminous Mixtures (L. F. Rader, chairman) has been actively engaged in the preparation of several new test methods as well as a review of existing methods. It has recommended the following new methods: (*1*) Proposed Tentative Method of Test for Resistance to Plastic Flow of Bituminous Mixtures by Means of Marshall Apparatus, (*2*) Proposed Tentative Method of Test for Resistance to Deformation and Cohesion of Bituminous Mixtures by Means of Hveem Apparatus, and (*3*) Tentative Method of Preparing Bituminous Mixtures by Means of California Kneading Compactor. The subcommittee is also considering the preparation of a recommended practice for design procedures using the test methods noted above.

The subcommittee has recommended certain modifications of Method D 1074 and has recommended several editorial revisions of Method D 1461.

Subcommittee B-3 on Distillation (J. W. Donegan, chairman) is engaged in a study of the repeatability and reproducibility of the Standard Method D 402 and, in view of the magnitude of some deviations, is considering means of improving test procedures through a cooperative testing program. It has made a study of a similar test, British Institute of Petroleum Method IP 27/56, which has indicated the desirability of certain editorial changes in D 402 which are now being prepared.

The subcommittee has recommended that the Tentative Method of Test for Water in Bituminous Materials (D 95 - 56 T) be adopted as standard.

Subcommittee B-5 on Softening Point (D. F. Fink, chairman) is initiating a cooperative testing program on a single thermometer/bath method for possible incorporation into Method D 36.

Subcommittee B-6 on Extraction and Recovery of Constituents from Bituminous Mixtures (E. W. Klinger, chairman) has established four task groups to make cooperative studies of a variety of extraction methods now in general use by various agencies. The purpose of this work is to evaluate the advantages and disadvantages of each of these methods as a basis for future activities of the subcommittee. It has recommended a revision of the ashing procedure in the Standard Method D 1097.

Subcommittee B-7 on Viscosity and Float Test (C. A. Benning, chairman) is engaged in the preparation of a method of test for the viscosity of tars and tar products by the Engler viscosimeter.

Subcommittee B-12 on Structural Properties of Mineral Aggregates (Stanton Walker, chairman) is working cooperatively with Subcommittee III-e of Committee C-9 on possible modifications of Method C 131. It is investigating the effect of changes in sample weight, as might result from the application of suggested tolerances, on measured loss in the test. Data on reproducibility and repeatability of the test method are also being accumulated. The subcommittee is also compiling a list of research subjects for submission to the Administrative Committee on Research.

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[5] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

Subcommittee B-14 on Specific Gravity (H. T. Williams, chairman) is working cooperatively with Subcommittee III-e of Committee C-9 on possible revisions of Methods C 127 and C 128. Studies are also under way on possible modifications of Method D 1188.

Subcommittee B-15 on Sampling Road Materials (R. E. Britton, chairman) has recommended revisions of Methods D 75. It is investigating an automatic, mechanical sampling device for use in securing representative samples of aggregates from plant bins.

Subcommittee B-16 on Setting Qualities of Bituminous Mixtures (A. B. Cornthwaite, chairman) is engaged in studies of various test methods for determining the curing rate of liquid asphalts. Studies are being made with the rolling-ball method, the Shell microviscometer and the Bureau of Public Roads thin film test.

Subcommittee B-17 on Emulsion Tests (P. E. McCoy, chairman) has established precision limits for tests included in Method D 244 and has recommended the incorporation of this material in Method D 244. Work has been initiated to develop a new method for testing MS emulsified asphalts and to modify and improve the current demulsibility test procedure. Efforts are being made to devise means of improving the viscosity test procedure.

Subcommittee B-18 on Loss on Heating (W. E. Spelshouse, chairman) has initiated studies to improve the accuracy of Method D 6. The studies are being directed primarily toward a method of calibrating ovens to give more consistent results.

Subcommittee B-19 on Accelerated Tests for Durability of Bituminous Materials (D. C. Taylor, chairman) is preparing a Method of Test for Determining the Effect of Heat and Air on Asphaltic Materials. It is also undertaking a cooperative study of a Method of Test for Aging Index of Bituminous Materials using the Shell microviscometer.

Subcommittee B-24 on Penetration Test (J. Y. Welborn, chairman) has completed a cooperative testing program to determine the precision of Method D 5, revised to provide closer control of the apparatus and procedure. Further revisions of the method are now under consideration and additional cooperative tests are being conducted.

Subcommittee B-26 on Effect of Water on Bituminous Coated Aggregates (W. H. Goetz, chairman) has prepared a Method of Test for Coating and Stripping of Bitumen-Aggregate Mixtures. Modifications of the method are now being made to overcome certain features objectionable to members of the subcommittee and of Committee D-4.

Subcommittee C-4 on Emulsified Asphalts (C. E. Proudley, chairman) is giving consideration to a number of proposed changes in Specifications D 977.

Subcommittee C-6 on Brick and Block Pavements (W. G. Doherty, chairman) has recommended the withdrawal of Specifications D 57, D 58, D 112, and D 132. Committee D-4 may wish to give further consideration to these recommendations but the recommendation for withdrawal is included in this report.

Subcommittee C-9 on Bituminous Penetration Macadam Surfaces and Bases (C. F. Parker, chairman) is considering possible modifications of Specification D 693.

Subcommittee C-10 on Plant Mix Bituminous Surfaces and Bases (Fred Hubbard, chairman) has recommended certain aggregate gradation changes in Specifications D 947 and D 1073. The subcommittee held a joint meeting with representatives of the American Association of State Highway Officials Committee on Materials and representatives of The Asphalt Institute to consider the possibility of achieving better consistency in bituminous mixture specifications of the three organizations. This meeting resulted in a number of suggested changes in the two standards listed above for consideration by the subcommittee.

Subcommittee D-1 on Calcium Chloride and Sodium Chloride for Use as Road Materials (H. F. Clemmer, chairman) has recommended a revision of Specification D 632.

Subcommittee D-2 on Highway Traffic Marking Materials (N. G. Smith, chairman) is making a study of various approaches to a performance type of specification for traffic paints.

Subcommittee D-3 on Joint Fillers and Asphalt Planks (H. F. Clemmer, chairman) is preparing modifications of Specification D 544. Present plans are to replace this specification with two specifications: one for joint fillers not containing bitumen and the other for joint fillers containing bitumen. The subcommittee is considering possible changes in Specification D 1190 and Method D 1191.

Joint Subcommittee of Committees D-4 and D-18 on Tests and Specifications for Stabilized Soils (H. F. Clemmer, chairman) has recommended the following proposed new methods: (*1*) Method of Test for Determining the Density of Soil-in-Place by the Sand-Cone Method, (*2*) Method of Test for Moisture-Density Relations of Soils Using a 10-lb Rammer and an 18-in. Drop, and (*3*) Method of Test for Determination of Moisture-Penetration Resistance Relations of Fine-Grained Soils. The subcommittee has also recommended further revisions of Method D 698.

In addition, the subcommittee has given consideration to tentatives under its jurisdiction and, pending further consideration of possible revisions, recommended continuation of the following tentatives: D 915, D 916, D 1241, and D 1411.

This report has been submitted to letter ballot of the committee, which consists of 170 members; 116 members returned their ballots, of whom 112 have voted affirmatively and 4 negatively.

Respectfully submitted on behalf of the committee,

K. B. WOODS,
Chairman.

JOHN M. GRIFFITH,
Secretary.

REPORT OF COMMITTEE D-5

ON

COAL AND COKE*

Committee D-5 on Coal and Coke held one meeting during the year: in Atlantic City, N. J., on June 20, 1957. A meeting of the Advisory Subcommittee was held in St. Louis, Mo., on February 11, 1958, and subcommittee meetings were held in Atlantic City in June, 1957 and in St. Louis, February 10 to 13, 1958.

No changes were made during the year in the membership of the committee, there being 63 voting members of whom 16 are classed as producers, 24 as consumers, and 23 as general interest.

The officers elected for the ensuing term of two years are as follows:

Chairman, O. W. Rees.

Vice-Chairman, W. M. Bertholf.

Secretary, R. L. Coryell.

Advisory Committee Members-at-Large: A. C. Fieldner, W. W. Anderson, C. C. Russell, C. H. Sawyer, and W. Selvig (Honorary Member).

A Task Group C, with Oscar Blatter as chairman, was authorized by the Advisory Subcommittee to carry out studies of the adiabatic calorimeter in comparison with the isothermal calorimeter.

ISO Activities. — Committee D-5, through the American Standards Association, has served as the official U. S. A. group participating in the development of international standards being conducted by Technical Committee 27, Solid Mineral Fuels, of the International Organization for Standardization. Effective participation in this work has been seriously hampered by lack of funds to finance travel to meetings, especially meetings of working groups, and by inability to secure data needed to evaluate the many proposals submitted for decision. In spite of the above mentioned difficulties, members of Committee D-5 have reviewed many ISO Documents and have developed replies to nine documents in connection with the work of ISO/TC 27. Ways and means of making our participation in this important work more effective are under consideration. This includes an effort which is being made to send delegates to the next meeting of ISO/TC 27 to be held at Harrogate, England, June 12 to 18, 1958.

Considerable progress has been made by this International Committee during the past year. Of particular interest is the fact that five important methods have reached the status of Draft ISO Recommendations and have been submitted to participating members for votes by the General ISO Secretariat. Recommended replies have been developed by the D-5 Advisory Subcommittee and submitted to the American Standards Association for transmittal to the General ISO Secretariat in Geneva, Switzerland.

REVISION OF STANDARD AND REVERSION TO TENTATIVE

The committee recommends revisions as appended hereto[1] of the Standard Method of Test for Cubic Foot

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*** Sixty-first Annual Meeting of the Society, June 22–27, 1958.**

[1] The revised Method of Test appears in the **1958 Book of ASTM Standards, Part 8.**

Weight of Crushed Bituminous Coal (**D 291 – 29**)[2] and reversion of the method to tentative.

ADOPTION OF TENTATIVE REVISIONS AS STANDARD

The committee recommends that the tentative revisions relating to sections on carbon and hydrogen, and nitrogen of the Standard Methods of Laboratory Sampling and Analysis of Coal and Coke (**D 271 – 48**)[2] be approved for reference to letter ballot of the Society with the following revisions:

Section 34 (*c*) (*1*).—Revise to read "*Water absorbent.* Anhydrous magnesium perchlorate of approximately 8 to 45 mesh size (Note 2)."

Section 36.—In Paragraph (*a*), delete the rest of the sentence following the words "weighed to the nearest 0.1 mg."

Revise Paragraph (*b*) to read as follows:

> (*b*) *Sample.*—Approximately 0.2 g (weighed to the nearest 0.1 mg) of air-dry sample ground to pass a 250-micron (No. 60) sieve shall be weighed into a combustion boat (Note 6).

New Note 6.—Add a new Note 6 following Section 36 to read as follows, renumbering subsequent Notes accordingly:

> *Note 6.*—It may be beneficial to grind fly ash, pit ash, calcined coke, and high mineral content materials to 100 mesh.

In the next to the last sentence of Paragraph (*c*), delete the rest of the sentence following the words "and finally weigh them to the nearest 0.1 mg."

Section 37.—Revise Note 2 following this section to read as follows: "Trade names of the reagent are: Anhydrone and Dehydrite."

REAPPROVAL OF STANDARDS

The committee recommends the reapproval of the following standards which have stood for six or more years without revision:

Standard Specifications for:

Gas and Coking Coals (**D 166 – 24**),
Classification of Coals by Rank (**D 388 – 38**), and
Classification of Coals by Grade (**D 389 – 37**).

Standard Methods of:

Drop Shatter Test for Coke (**D 141 – 48**),
Test for Volume of Cell Space of Lump Coke (**D 167 – 24**),
Sampling and Fineness Test of Powdered Coal (**D 197 – 30**),
Test for Cubic Foot Weight of Coke (**D 292 – 29**),
Test for Sieve Analysis of Coke (**D 293 – 50**),
Tumbler Test for Coke (**D 294 – 50**),
Test for Size of Anthracite (**D 310 – 34**),
Test for Sieve Analysis of Crushed Bituminous Coal (**D 311 – 30**),
Sampling Coke for Analysis (**D 346 – 35**),
Test for Grindability of Coal by the Hardgrove-Machine Method (**D 409 – 51**),
Test for Screen Analysis of Coal (**D 410 – 38**),
Designating the Size of Coal from Its Screen Analysis (**D 431 – 44**),
Drop Shatter Test for Coal (**D 440 – 49**),
Tumbler Test for Coal (**D 441 – 45**),
Sampling Coals Classed According to Ash Content (**D 492 – 48**), and
Test for Index of Dustiness of Coal and Coke (**D 547 – 41**).

Standard Definition of:

Terms Relating to Coal and Coke (**D 121 – 30**),
Terms Gross Calorific Value and Net Calorific Value of Solid and Liquid Fuels (**D 407 – 44**), and
Commercial Varieties of Bituminous and Sub-Bituminous Coals (**D 493 – 39**).

TENTATIVES CONTINUED WITHOUT REVISION

The committee recommends the continuation without revision of all other tentatives and tentative revisions of standards under its jurisdiction.

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[3]

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[2] 1955 Book of ASTM Standards, Part 5.

[3] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee II on Nomenclature and Definitions (C. H. Sawyer, chairman) continued its review of terms used in standards and is referring such terms that appear to be ambiguous to appropriate subcommittees or other committees of the Society. A suitable procedure for defining difficult terms is under consideration.

Subcommittee VII on Pulverizing Characteristics of Coal (H. F. Yancey, chairman) considered further the revision of the Standard Method of Test for Grindability of Coal by the Hardgrove-Machine Method (D 409 - 51) in which it was proposed that suitable uniform materials of constant indexes could be found among the new industrial chemicals and materials. A study of the grindability of 40 samples was presented in which tests were made on coal of the standard 16-30 sieves and also on 16-60 sieves. Consideration is being given to a revision of the standard to include the expanded particle-size range. Modification of the procedure was also considered so that results of better reproducibility can be obtained on high-moisture sub-bituminous coal and lignite.

Subcommittee XV on Plasticity and Swelling of Coal (Michael Perch, chairman) considered the use of an electric furnace as an alternate to the gas burner in the Standard Method of Test for Free-Swelling Index of Coal (D 720 - 57). A description of the apparatus and a comparative study of the accuracy of the method with the standard was requested.

The third draft of a proposed standard method of testing coal by the Gieseler plastometer was presented to the subcommittee. Further changes were suggested for incorporation and the subcommittee voted to submit the proposed method to letter ballot of the subcommittee for recommendation of advancement to tentative status.

Subcommittee XX on Sampling and Fineness Test of Pulverized Coal (E. L. Rogers, chairman) has planned tests to be carried out on various types of sampling tips and also for comparative testing of the standard cyclone and filter bags for collecting of samples. The subcommittee was of the opinion that it would recommend a separate standard for a method of calibrating screens. As a result of the various suggestions and tests, the subcommittee proposed issuing a revision of the tentative draft of Method D 197.

Subcommittee XXI on Methods of Analysis (R. F. Abernethy, chairman) approved by letter ballot a recommendation to Committee D-5 to advance the tentative revisions of methods of analysis for carbon, hydrogen, and nitrogen, and estimation of oxygen of Method D 271 to standard status. It recommended to continue in tentative status the test for equilibrium moisture in order to investigate the effect of less massive equipment on the final results. Sufficient investigations were completed on new test methods for the sulfur in ash, mineral carbonates, and fusibility of coal ash in oxidizing atmosphere to prepare proposed procedures. Proposed test procedures were prepared for revision of the present standards for fusibility of ash in a reducing atmosphere and volatile matter by a modified method. Further work is under way for new test methods for chlorine and forms of sulfur.

Subcommittee XXII on Physical Tests of Coke (B. P. Mulcahy, chairman) has under consideration revision of the Standard Method of Drop Shatter Test for Coke (D 141 - 48). Other methods for testing coke are to be likewise reviewed.

Subcommittee XXIII on Sampling (W. M. Bertholf, chairman).—Data have been assembled and the subcommittee can now write preliminary proposals on specifications for mechanical sampling problems. A draft proposal for preparation of coal samples is in the early stages of writing. No definite progress on coke sampling can be reported, although this subject is being considered.

This report has been submitted to letter ballot of the committee, which consists of 63 voting members; 56 members returned their ballots, of whom 51 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

O. W. REES,
Chairman.

C. C. RUSSELL,
Secretary.

REPORT OF COMMITTEE D-6

ON

PAPER AND PAPER PRODUCTS*

Committee D-6 on Paper and Paper Products held two meetings during the year: on June 20, 1957, in Atlantic City, N. J., and on February 21, 1958, in New York, N. Y.

Joint work between Committee D-6 and committees of the Technical Association of the Pulp and Paper Industry is progressing. Joint groups are working on methods for basis weight, tensile strength, bursting strength, flammability of treated papers, absorption by bibulous papers, and sampling. Task groups are being organized to work on a recommended practice for interlaboratory evaluation of test methods for paper and paper products, and quantitative methods for determining mineral fillers and coating constituents in paper.

A Symposium on New Developments With Accompanying Requirements for New Testing Methods, consisting of the following papers, is being sponsored by the Committee at the 1958 Annual Meeting of the Society:

"Some Historical Developments in Paper Testing," by W. R. Willets and F. R. Marchetti.

"Testing of Synthetic Fiber Papers," by F. H. Koontz and J. K. Owens.

"Technology of Non-Woven Structures: Paper and Textile Processes and End Uses," by J. T. Taylor and F. J. McLaughlan.

"New Developments in the Internal Bonding of Paper," by K. W. Britt.

"Paper Base for Laminates in Printed Circuitry," by N. A. Skow.

"Polyethylene Extrusion Coated Papers—An Appraisal of Testing Methods," by K. Thompson.

"New Approaches in Packaging Material Testing for the Food Industry," by P. K. Wolker.

"Knit Paper—Its Uses as a Utilitarian Textile," by R. H. Marks.

The committee consists of 80 members, of whom 31 are classified as producers, 18 as consumers, and 31 as general interest members.

The officers and Members-at-Large on the Advisory Committee elected for the ensuing term of two years are as follows:

Chairman, R. H. Carter.

Vice-Chairman, H. A. Birdsall.

Secretary, R. E. Green.

Advisory Committee Members-at-Large: K. A. Arnold and C. E. Brandon.

REVERSION OF STANDARD TO TENTATIVE

The committee recommends the reversion to tentative of Standard Methods of Test for Thickness of Paper and Paper Products (**D 645 – 43**)[1] since the committee contemplates a thorough revision of the method during the coming year.

ADOPTION OF TENTATIVES AS STANDARD WITHOUT REVISION

The committee recommends that the following tentatives be approved for reference to letter ballot of the Society for adoption as standard without revision:

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* Sixty-first Annual Meeting of the Society, June 22–27, 1958.

[1] 1955 Book of ASTM Standards, Part 7.

Tentative Method of Test for:

- Resistance of Paper to Passage of Air (**D 726 – 55 T**),[1]
- Water Resistance of Paper, Paperboard, and Other Sheet Materials by the Dry-Indicator Method (**D 779 – 55 T**),[1] and
- Pinholes in Glassine and Other Greaseproof Papers (**D 1221 – 52 T**).[1]

REVISION OF STANDARDS, IMMEDIATE ADOPTION

The committee recommends for immediate adoption revisions as appended hereto[2] of the Standard Method of Test for Titanium Dioxide in Paper (**D 921 – 49**),[1] and accordingly asks for the necessary nine-tenths affirmative vote at the Annual Meeting in order that the revisions may be referred to letter ballot of the Society.

WITHDRAWAL OF TENTATIVE

The committee recommends the withdrawal of the Tentative Methods of Test for Water Vapor Permeability of Paper and Paperboard (**D 988 – 51 T**).[1] The Tentative Methods of Test for Measuring Water Vapor Transmission of Materials in Sheet Form (**E 96 – 53 T**)[1] will be used in place of the withdrawn method.

REAPPROVAL OF STANDARDS

The committee recommends the reapproval of the following standards which have stood for six or more years without revision:

Standard Methods of:

- Test for Machine Direction of Paper (**D 528 – 41**),
- Test for Paraffin Content of Waxed Paper (**D 590 – 42**),
- Test for Folding Endurance of Paper (**D 643 – 43**),
- Qualitative Examination of Mineral Filler and Mineral Coating of Paper (**D 686 – 48**),
- Test for Pentosans in Paper (**D 688 – 44**),
- Turpentine Test for Grease Resistance of Paper (**D 722 – 45**),
- Test for Hydrogen Ion Concentration (pH) of Buffered Paper Extracts (**D 778 – 50**),
- Test for Printing Ink Permeation of Paper (Castor Oil Test) (**D 780 – 46**),
- Test for Blocking Resistance of Paper and Paperboard (**D 918 – 49**),
- Test for Organic Nitrogen in Paper and Paperboard (**D 982 – 52**),
- Test for Paraffin Wax Absorptiveness of Paper (**D 983 – 50**),
- Test for Reducible Sulfur in Paper (**D 984 – 57**),
- Preparation of a Magnesium Oxide Standard for Spectral Reflectivity (**D 986 – 50**),
- Creasing Paper for Permeability Tests (**D 1027 – 51**), and
- Test for Water-Soluble Sulfates in Paper and Paperboard (**D 1099 – 52**).

TENTATIVES CONTINUED WITHOUT REVISION

The committee recommends the continuation without revision of all other tentatives under the jurisdiction of the committee.

EDITORIAL CHANGES

The committee recommends editorial changes as indicated in the following standards:

Standard Method of Test for Paraffin Content of Waxed Paper (D 590 – 42):[1]

Section 2.—Delete this entire section and renumber subsequent sections accordingly.

Standard Method of Test for Pentosans in Paper (D 688 – 44):[1]

New Section. –Add a new Section 2 on Significance of Test to read as follows, renumbering the present Section 2 and subsequent sections accordingly:

> 2. Pentosans in paper are part of the noncellulosic carbohydrates included in the terms "hemi-celluloses" or polysaccharides and consist chiefly of hylan and araban.
>
> The effect of the pentosans on the chemical or physical properties of paper is not definitely known at present. Quantitative measurements may be informative, especially if the amount of pentosans in the pulp from which the paper was made is known.

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[2] The revised standard appears in the 1958 Book of ASTM Standards, Part 6.

Method of Test for Paraffin Wax Absorptiveness of Paper (D 983 – 50):[1]

New Section.—Add a new Section 2 on Significance of Test to read as follows, renumbering the present Section 2 and subsequent sections accordingly:

> 2. This method measures the amount of wax required to saturate a paper without producing any surface film. The results may be used for comparing two or more samples to judge their suitability for wet waxing, dry waxing, or laminating, or to judge the performance of saturating machines. The method is of chief interest to converters or laminators as a screening test or for control purposes.

Standard Method of Test for Reducible Sulfur in Paper (D 984 – 54):[1]

New Section.—Add a new Section 2 on Significance of Test to read as follows, renumbering the present Section 2 and subsequent sections accordingly:

> 2. This method is not suitable in all cases for determining whether a given paper will tarnish polished metals, especially silver.
>
> Paper that, when tested by this method, shows less than 0.0008 per cent reducible sulfur probably can be assumed to be non-tarnishing as far as sulfur is concerned. Results showing more than 0.0008 per cent reducible sulfur do not necessarily indicate that the paper will cause tarnishing. Likewise, the results of the test give no information regarding the distribution of the sulfur in the paper.
>
> When results showing more than 0.0008 per cent reducible sulfur are obtained, or information regarding the distribution of the sulfur or the presence of other materials causing stains on polished silver is desired, the paper should be evaluated by an accelerated tarnishing test.

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[3]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Sampling and Conditioning (T. W. Lashof, chairman) is currently evaluating three approaches to the problem of sampling in the paper industry. The committee has reviewed the sampling procedures proposed by the International Standards Organization, Technical Committee 6. Recommended practices for maintaining conditioning rooms for paper testing and the effects of conditioning on paper are being studied.

Subcommittee II on Chemical Test Methods (A. P. Tracy, chairman) is initiating a new study of a method of determining mold resistance of paper.

Subcommittee IV on Physical Test Methods for Container Board (W. B. Lincoln, Jr., chairman) is restudying the original work on the flat crush test. A questionnaire will be sent out to interested members in order to design a revision of Method D 1225; a cooperative test will be based on the results of this information. In cooperation with TAPPI, improvement of the Elmendorf method of testing cylinder boards is under study. Improved designs of the Mullen tester for use with corrugated board is being undertaken.

Subcommittee VI—Editorial (L. S. Reid, chairman) is continuing the preparation of significance of test statements for all new and revised methods.

This report has been submitted to letter ballot of the committee, which consists of 80 voting members; 54 members returned their ballots, of whom 46 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

R. H. CARTER,
Chairman.

R. E. GREEN,
Secretary.

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[3] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

REPORT OF COMMITTEE D-7
ON
WOOD*

Committee D-7 on Wood and seven of its subcommittees met in St. Louis, Mo. on February 10 and 11, 1958. Meetings of the following subcommittees preceded the main committee meeting: Subcommittee I on Specifications for Timber; Subcommittee IV on Wood Paving Blocks; Subcommittee V on Methods of Preservative Treatment of Timber; Subcommittee VI on Timber Preservatives; Subcommittee VII on Wood Poles and Cross Arms; Subcommittee XIII on Durability and Exposure; and Subcommittee XV on Wood-Base Fiber and Particle Panel Materials.

Fifteen new members were elected to the committee; there were nine resignations. One new consulting member was appointed, and two committee members were appointed to represent D-7 on Committees E-1 and D-20.

The officers elected for the ensuing term of two years are as follows:

Chairman, L. J. Markwardt.
Vice-Chairman, L. W. Wood.
Secretary, L. W. Smith.

ISO Proposal on Fiberboards.—Last year it was reported that the American Standards Association had received a proposal originating in Norway to form an international committee on fiberboards under the auspices of the International Standardization Organization. The scope as indicated was for the purpose of facilitating international trade and to establish international agreement on standardization of specifications—including terminology, dimensions and tolerances, designations, rules for sampling and methods of tests—for fiber building board. Although ASA, in concurrence with the recommendation of D-7, advised against the establishment of this project, word has been received that the committee has now been organized by vote of a number of other nations participating in ISO work. Consideration should now be given as to whether fiberboard interests in the United States wish to participate in any way. Even if no positive action is taken, ASA serves as an observer to report on the future activity of the ISO panel.

NEW TENTATIVE

The committee recommends for publication as tentative the Definitions of Terms Relating to Wood-Base Fiber and Particle Panel Materials as appended hereto.[1]

ADOPTION OF TENTATIVE AS STANDARD WITH REVISIONS

The committee recommends that the Tentative Methods of Static Tests of Wood Poles (**D 1036 - 55 T**)[2] be approved for reference to letter ballot of the Society for adoption as standard with revisions as indicated.

Preface.—In lines 7 and 8 change the word "top" to "tip."

Section 9 (2).—Delete the word "exact."

Section 9 (4).—Change the word "top"

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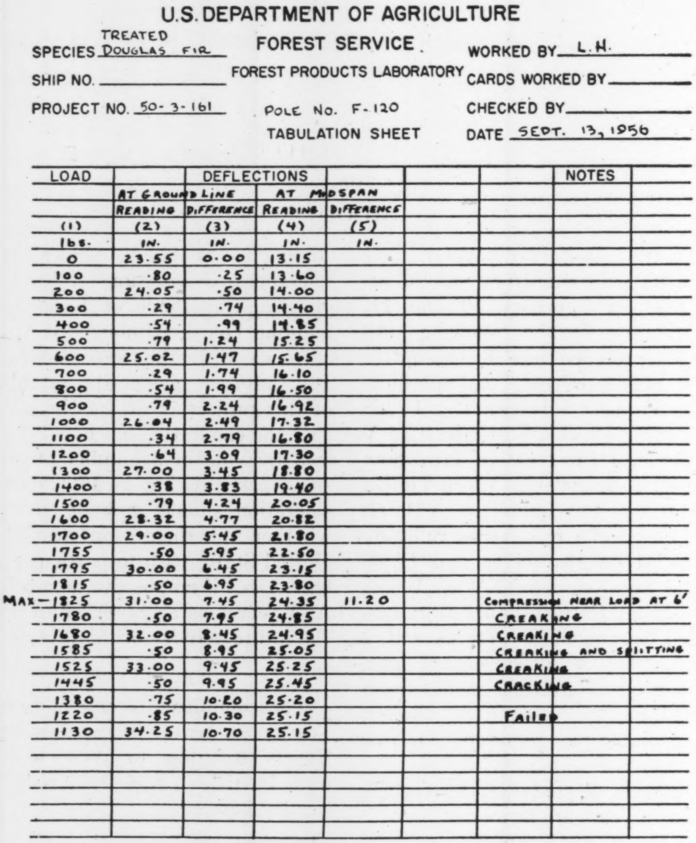
* Sixty-first Annual Meeting of the Society, June 22–27, 1958.

[1] The new and revised tentatives appear in the 1958 Book of ASTM Standards, Part 6.

[2] 1955 Book of ASTM Standards, Part 4.



U.S. DEPARTMENT OF AGRICULTURE
FOREST SERVICE
FOREST PRODUCTS LABORATORY

SPECIES TREATED DOUGLAS FIR
SHIP NO. ____
PROJECT NO. 50-3-161
POLE NO. F-120
TABULATION SHEET
WORKED BY L. H.
CARDS WORKED BY ____
CHECKED BY ____
DATE SEPT. 13, 1956

LOAD		DEFLECTIONS					NOTES	
	AT GROUND LINE		AT MIDSPAN					
	READING	DIFFERENCE	READING	DIFFERENCE				
(1)	(2)	(3)	(4)	(5)				
lbs.	IN.	IN.	IN.	IN.				
0	23.55	0.00	13.15					
100	.80	.25	13.60					
200	24.05	.50	14.00					
300	.29	.74	14.40					
400	.54	.99	14.85					
500	.79	1.24	15.25					
600	25.02	1.47	15.65					
700	.29	1.74	16.10					
800	.54	1.99	16.50					
900	.79	2.24	16.92					
1000	26.04	2.49	17.32					
1100	.34	2.79	16.80					
1200	.64	3.09	17.30					
1300	27.00	3.45	18.80					
1400	.38	3.83	19.40					
1500	.79	4.24	20.05					
1600	28.32	4.77	20.82					
1700	29.00	5.45	21.80					
1755	.50	5.95	22.50					
1795	30.00	6.45	23.15					
1815	.50	6.95	23.80					
MAX—1825	31.00	7.45	24.35	11.20		COMPRESSION NEAR LOAD AT 6′		
1780	.50	7.95	24.85			CREAKING		
1680	32.00	8.45	24.95			CREAKING		
1585	.50	8.95	25.05			CREAKING AND SPLITTING		
1525	33.00	9.45	25.25			CREAKING		
1445	.50	9.95	25.45			CRACKING		
1380	.75	10.20	25.20					
1220	.85	10.30	25.15			Failed		
1130	34.25	10.70	25.15					

FIG. 1.—Tabulation of Data Observed in Machine Test.

to "tip." Add at the end "to the nearest $\frac{1}{16}$ in."

Section 10(b).—Revise the first sentence to read as follows:

> A support *B* shall be provided at a point about three-quarters of the distance from the ground line to the point of load application to minimize vertical movement at that point and reduce the stress from the weight of the pole.

In the last sentence, delete the word "top" after the word "pole."

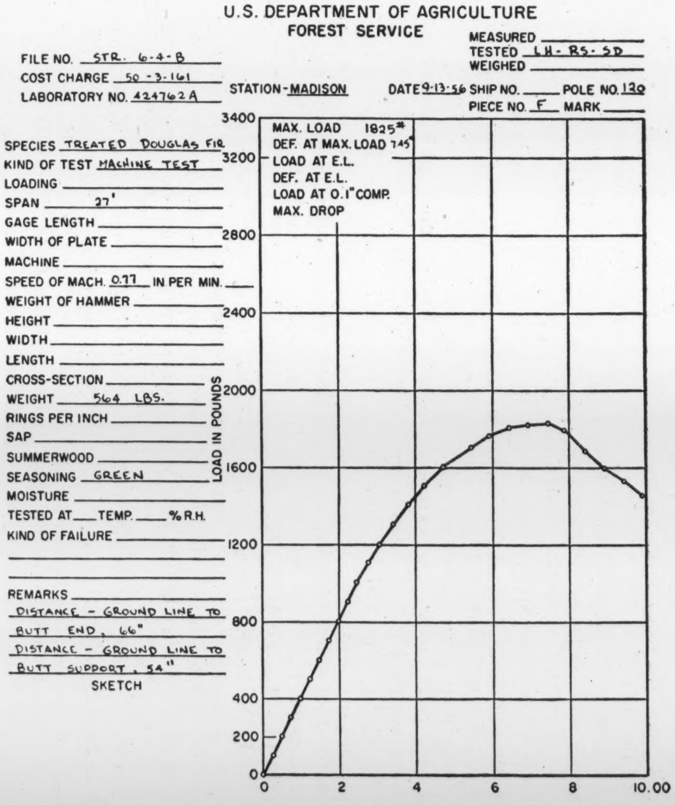
Section 11.—In the first sentence change "below the top" to read "from the tip."

Revise the second sentence to read "The pulling line shall be kept level between the winch position and the point where load is applied to the pole."

In the definition of C_t following the formula, change "circumference at point of load" to read "circumference at ground line."

Section 15(a).—In the last sentence, change the words "top of the pole" to read "load point."

Section 16(a) (1).—Change the words "top deflection" to read "deflection of the load point."



U.S. DEPARTMENT OF AGRICULTURE
FOREST SERVICE

FILE NO. STR. 6-4-B
COST CHARGE 50-3-161
LABORATORY NO. 424762A
STATION-MADISON
DATE 9-13-56
MEASURED
TESTED LH-RS-SD
WEIGHED
SHIP NO.
POLE NO. 120
PIECE NO. F
MARK

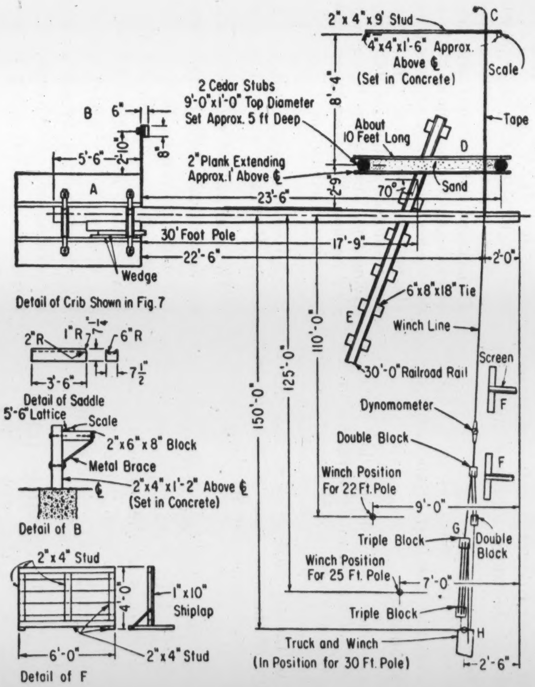
SPECIES TREATED DOUGLAS FIR
KIND OF TEST MACHINE TEST
LOADING
SPAN 27'
GAGE LENGTH
WIDTH OF PLATE
MACHINE
SPEED OF MACH. 0.77 IN PER MIN.
WEIGHT OF HAMMER
HEIGHT
WIDTH
LENGTH
CROSS-SECTION
WEIGHT 564 LBS.
RINGS PER INCH
SAP
SUMMERWOOD
SEASONING GREEN
MOISTURE
TESTED AT ___ TEMP. ___ % R.H.
KIND OF FAILURE

REMARKS
DISTANCE - GROUND LINE TO BUTT END, 66"
DISTANCE - GROUND LINE TO BUTT SUPPORT, 54"
SKETCH

MAX. LOAD 1825#
DEF. AT MAX. LOAD 7.45"
LOAD AT E.L.
DEF. AT E.L.
LOAD AT 0.1" COMP.
MAX. DROP

LOAD IN POUNDS
3400
3200
2800
2400
2000
1600
1200
800
400
200
0
0
2
4
6
8
10.00

FIG. 2.—Load-Deflection Curve and Related Data in Machine Test.



2"x4"x9' Stud
C
4"x4"x1'-6" Approx. Above ℄ (Set in Concrete)
Scale
8'-4"
2 Cedar Stubs 9'-0"x1'-0" Top Diameter Set Approx. 5 ft Deep
About 10 Feet Long
Tape
B
6"
2'-10"
8"
5'-6"
2" Plank Extending Approx. 1' Above ℄
D
Sand
2'-9"
70°
A
23'-6"
30' Foot Pole
17'-9"
22'-6"
2'-0"
Wedge
Detail of Crib Shown in Fig. 7
6"x8"x18" Tie
E
Winch Line
2"R
1"R
1"
6"R
3'-6"
7½"
Detail of Saddle
30'-0" Railroad Rail
Screen
F
150'-0"
125'-0"
110'-0"
5'-6" Lattice
Scale
2"x6"x8" Block
Metal Brace
2"x4"x1'-2" Above ℄ (Set in Concrete)
Dynomometer
Double Block
Detail of B
Winch Position For 22 Ft. Pole
9'-0"
G
Triple Block
Double Block
2"x4" Stud
Winch Position For 25 Ft. Pole
7'-0"
4'-0"
1"x10" Shiplap
Triple Block
H
Truck and Winch (In Position for 30 Ft. Pole)
6'-0"
2"x4" Stud
2'-6"
Detail of F

FIG. 3.—A Field Layout Which Has Been Used for Pole Strength Tests.

Table II.—In line 3 and the second line of the footnote, change "top" to read "tip."

Section 17(d).—Add a second Explanatory Note at the end of the section to read as follows:

NOTE.—Since shear deformation makes a negligible contribution to the deflection of the pole in test, the modulus of elasticity calculated by the formula will correspond more nearly to the true (compression) modulus of elasticity of the wood than to the apparent (bending) modulus in the standard test of a small clear specimen.

Section 18(e).—Delete this section.

Section 20.—In Paragraph (*a*), in the explanation of a in the formula, change the word "top" to read "tip."

In Paragraph (*b*), delete the last sentence.

Section 21.—In Paragraph (*b*), in the first sentence, in the explanation of w in the formula, and in the first sentence of the Note change the words "above the load point" to read "from the load point to the tip end."

In Paragraph (*c*), at the end of the paragraph, add the same Explanatory Note as that added to Section 17(*d*).

Figures 4, 5, and 6.—New sample data sheets are shown for Figs. 4 and 5, as shown in the accompanying Figs. 1 and 2, to bring them into accordance with the revised text. Figure 6, as shown in the accompanying Fig. 3, is slightly revised for clarification and to bring it into accordance with the revision of Paragraph 2 of the Appendix. Revise the caption for Fig. 6 to read as follows: "A Field Layout Which Has Been Used for Pole Strength Tests."

Section 23(a).—In the first sentence change the words "below the ground line" to read "from the ground line toward the butt."

Appendix.—Revise the second paragraph to read as follows:

Figure 6 shows a field layout which has been used, with crib *A*, test pole, supporting rail *E*, pulling tackle *G*, and winch *H*. The sketch indicates winch positions for three lengths of poles. Details of parts of the testing apparatus are also included. Points *B* and *C* are reference points for measuring the movement of the ground line and the load point of the test pole, respectively. The sand and plank barrier at *D* is a safety device to protect against failure of the tackle or pulling line. The screens *F* are to protect the man reading the dynamometer from possible breaks in the pulling line.

REVISION OF STANDARD AND REVERSION TO TENTATIVE

The committee recommends revisions as appended hereto[1] and reversion to tentative of the Standard Specifications for Wood Paving Blocks for Exposed Pavements (**D 52 - 57**),[2] including a change in title to Standard Specifications for Wood Paving Blocks for Exposed Platforms, Pavements, Driveways, and Interior Floors Exposed to Wet and Dry Conditions.

REVISION OF STANDARDS, IMMEDIATE ADOPTION

The committee recommends for immediate adoption revisions as indicated of the following standards, and accordingly asks for the necessary nine-tenths affirmative vote at the Annual Meeting in order that the revisions may be referred to letter ballot of the Society:

Standard Specifications for Round Timber Piles (D 25 - 55):[2]

Section 4(c).—Add the following sentence to the end of the paragraph: "Requirements for spacing of knots shall be applied at the centers of the knots considered."

Section 6(a).—Add the following sentence: "Requirements for tip circumference of piles that are longer than the required length may be applied at the tip end of the required length."

Table I.—Change line 3 of the column headed "Length, ft" from "51 to 70" to read "55 to 70;" change line 4 of the same column from "71 to 90" to read

"75 to 90." Revise Footnote *a* to read as follows:

[a] In class B piles, a minimum circumference of 34 in. or diameter of 11 in. at a point 3 ft from the butt may be specified for lengths of 25 ft and under. In class C piles, a minimum circumference of 31 in. or diameter of 10 in. at a point 3 ft from the butt may be specified for lengths of 25 ft and under.

Section 12.—Revise to read as follows: "Spiral grain shall not exceed one-half of a complete twist in any 20 ft of length."

Section 17.—Revise to read as follows: "Spiral grain shall not exceed one complete twist in any 20 ft of length."

Standard Method of Test for Water in Creosote (D 370 - 33):[2]

Section 5.—Change the note at the end of this section to read as follows:

NOTE.—Method D 95 - 56 T is the preferred method where the determination of the water only is desired and the dehydrated material is not to be used subsequently for analysis."

Standard Method of Test for Integrity of Glue Joints in Laminated Wood Products for Exterior Service (D 1101 - 53):[2] Revise as appended hereto.[1]

REAPPROVAL OF STANDARDS

The committee recommends the reapproval of the following standards which have stood for six years without revision:

Standard Methods of Test for:

Coke Residue of Creosote (**D 168 - 30**),
Distillation of Creosote (**D 246 - 49**),
Benzene-Insoluble Matter in Creosote (**D 367 - 49**),
Specific Gravity of Creosote (**D 368 - 33**),
Specific Gravity, 38/15.5 C., of Creosote Fractions (**D 369 - 33**),
Tar Acids in Creosote and Creosote-Coal Tar Solutions (**D 453 - 41**), and
Veneer, Plywood, and Other Glued Veneer Constructions (**D 805 - 52**).

Standard Methods of:

Sampling and Testing Creosote (**D 38 - 33**),
Testing Small Clear Specimens of Timber (**D 143 - 52**),
Static Tests of Timbers in Structural Sizes (**D 198 - 27**),
Chemical Analysis of Zinc Chloride (**D 199 - 50**),
Chemical Analysis of Chromated Zinc Chloride (**D 1033 - 50**), and
Chemical Analysis of Tanalith (**D 1035 - 50**).

Standard Specifications for:

Creosote (**D 390 - 53**),
Creosote-Coal Tar Solution (**D 391 - 53**),
Zinc Chloride (**D 432 - 50**),
Chromated Zinc Chloride (**D 1032 - 50**), and
Tanalith (**D 1034 - 50**).

Standard Definitions of:

Terms Relating to Timber (**D 9 - 30**),
Terms Relating to Timber Preservatives (**D 324 - 41**), and
Terms Relating to Veneer and Plywood (**D 1038 - 52**).

Miscellaneous:

Volume and Specific Gravity Correction Tables for Creosote and Coal Tar (**D 347 - 33**), and
Nomenclature of Domestic Hardwoods and Softwoods (**D 1165 - 52**).

TENTATIVES CONTINUED WITHOUT REVISION

The committee recommends the continuation without revision of the following standards:

Tentative Methods of Test for:

Alpha-Cellulose in Wood (**D 1103 - 55 T**), and
Methoxyl Groups in Wood and Related Materials (**D 1166 - 55 T**).

Tentative Method of Testing:

Wood Preservatives by Laboratory Soil-Block Cultures (**D 1413 - 56 T**).

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[3]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Specifications for Timber (L. W. Wood, chairman).—Activities of the subcommittee during the year have included the revision of the Standard Specifications for Round Timber Piles (D 25 – 55) for publication in the 1958 Book of Standards. Proposed revisions of Tentative Methods for Establishing Structural Grades of Lumber (D 245 – 57 T) are under consideration. One of the complicated problems involved is that relating to basic stresses for Douglas fir, which is now being studied by the Forest Products Laboratory on the basis of extensive sampling of the material throughout its range. Studies on basic stresses for structural lumber are continuing.

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[3] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

Subcommittee II on Laminated Timber (Frank J. Hanrahan, chairman).—Research has continued since the first standard on tests for the evaluation of the integrity of glue joints in laminated wood products for exterior use was developed some years ago. Special consideration has been given to the reduction in over-all time required for completion of evaluation by the present method. A revised procedure recommended for adoption, as reported elsewhere, incorporates changes that effect a reduction of the immersion period from 8 to 2½ hr, making it essentially a two-cycle test rather than a three-cycle procedure. This reduces the total time required for this test from 12 to 8 days.

Subcommittee IV on Wood Paving Blocks (Howard O'Brien, chairman) revised the Standard Specifications for Wood Paving Blocks for Exposed Pavements (D 52 – 57). The principal revision concerns a change in requirements with respect to rate of growth for southern yellow pine and Douglas fir to not less than four annual rings per inch measured over a 3-in. portion on a representative radial line. A tolerance permitting not more than 5 per cent of the pieces in any lot to have 3 rings per in. is included. The revision unifies the rate of growth requirements for the two species.

Subcommittee V on Methods of Preservative Treatment of Timber (C. W. Best, chairman).—Since the reactivation of the subcommittee under the present chairman, work has been started on the development of specifications for the preservative treatment of timber products. This work will be continued during the year with the objective of having a tentative prepared for approval during the coming year. The specifications are being correlated with those of the American Wood-Preservers' Association.

Subcommittee VI on Timber Preservatives (D. L. Davies, chairman).—A review of some of the present standards indicates that some of the references in the footnote are now out of date and therefore they are being corrected editorially.

Subcommittee VII on Wood Poles and Cross Arms (R. P. A. Johnson, chairman).—The principal activity of Subcommittee VII continues to be the ASTM Wood Pole Research Program. The Technical Advisory Group in the Subcommittee, with responsibility for conducting the program and reviewing reports, held one meeting in March 1957 and has also devoted a great deal of time and thought to the many and complex technical questions involved.

The program now nears completion. Nine interim reports have been distributed by ASTM, with two additional reports in process. A final report will then be prepared for publication and general distribution which will not only summarize and interpret the data from the program but also report related research which will be of value to American Standards Association Sectional Committee O5 in reconsidering the whole subject of specifications and dimensions for wood poles.

Two special problems have recently received attention. The problem of strength values in lodgepole pine poles was fully reconsidered with the aid of a specific gravity survey conducted by Bell Telephone Laboratories, and a supplemental interim report was issued.

The problem of strength of treated southern pine poles was considered and it was agreed that it should be possible to obtain satisfactory treatment with less effect upon strength than was observed in the southern pine tests. To determine if this can be done, additional research along two lines is now under way. Experiments are being made at the treating plant and test material is under treatment to see if milder schedules will insure adequate penetration and distribution of preservative and to observe the effects on strength.

The subcommittee has made minor revisions in Tentative Methods of Static Tests of Wood Poles (D 1036 - 55 T) which is being recommended for advancement to standard.

Further study has been made of the standard method of test of strength for wood cross arms. It is recognized that two methods may be used: (1) a simple beam test, which gives a modulus-of-rupture or fiber-stress value that is useful in specification or design of cross arms; and (2) a structural test with the arm and braces fastened to a pole and load applied at each pinhole, which gives comparative load values useful for comparisons between structures or for market development. The second method is not readily standardized because of the variety of structures possible. Subcommittee VII believes, however, that the first method should be standardized, and has taken action leading to reactivation of a task group to undertake this work.

Subcommittee IX on Methods of Testing (L. J. Markwardt, chairman).—Consideration will be given during the year to the development of two test methods relating to the evaluation of two products employed in heavy timber construction, namely, a method of test for metal connectors employed in timber joints and fastenings, and a method of testing joist hangers. Consideration will be given also to the development of a test method for ladders.

Subcommittee X on Nomenclature and Definitions (R. R. Cahal, chairman).—A number of editorial changes were made by the subcommittee in the Nomenclature for Domestic Hardwoods and Softwoods (D 1165 - 52) to bring them in line with the current check list as intended, but no further work is presently under way.

Subcommittee XIII on Durability and Exposure (R. M. Lindgren, chairman).—Two meetings were held during the year, the first in Cleveland, Ohio, on October 1, 1957, and the second on February 10, 1958 in St. Louis. Considerable progress was made in discussing possible revisions of the Tentative Method of Testing Wood Preservatives by Laboratory Soil-Block Cultures (D 1413 - 56 T), and in reaching agreement on the content of a new standard to cover accelerated field tests of preservatives in $\frac{3}{4}$-in. stakes. It was the consensus of the committee that attempts at revision at this time of Method D 1413 - 56 T would be premature and, therefore, that this tentative should continue in its present form and status for the time being.

Five task-force groups set up within the subcommittee to consider and develop a new standard on accelerated field tests of preservatives were very productive through the year. As a result of these efforts, it now appears likely that a draft of a possible tentative standard will be ready for consideration by the entire subcommittee in the late summer or early fall.

Subcommittee XV on Wood-Base Fiber and Particle Panel Materials (W. C. Lewis, chairman).—A subject that has had much discussion and consideration is that relating to definitions of terms for fiber and particle board materials. After several years' work a draft has been developed that meets general approval, and is recommended for adoption under the title, "Tentative Definitions of Terms Relating to Wood-Base Fiber and Particle Panel Materials."[1]

Work has continued in the development of additional tests for fiberboards and particle boards. The committee recommends that Method D 1037 be continued as tentative but that the title be changed to "Tentative Methods of Test for Evaluating the Properties of Wood-Base Fiber and Particle Panel Materials." Additional test procedures as follows: (1) falling ball impact tests, (2) navy-type abrasion test, and (3) direct screw withdrawal are being considered by the subcommittee and will be included in a subsequent revision.

Methods of test and a product specification for insulated roof deck slabs are being studied, as well as the possible need for a particle board standard.

Subcommittee XVI on Wood Flour (H. W. Shader, chairman).—Following a conference in Atlantic City at the time of the Annual Meeting in June 1957, arrangements were made to organize a subcommittee on wood flour for the purpose of developing specifications and methods of test that will establish more definite procedures between buyer and seller. It was brought out that a number of problems relating to specifications and methods of sieve analysis required further study and that ASTM provided a most suitable mechanism for this work. The subcommittee is now being organized on a national scope.

This report has been submitted to letter ballot of the committee, which consists of 96 voting members; 76 members returned their ballots, of whom 71 have voted affirmatively and 2 negatively.

Respectfully submitted on behalf of the committee,

L. J. MARKWARDT,
Chairman.

L. W. SMITH,
Secretary.

REPORT OF COMMITTEE D-8

ON

BITUMINOUS WATERPROOFING AND ROOFING MATERIALS*

Committee D-8 on Bituminous Waterproofing and Roofing Materials held two meetings during the year: in Atlantic City, N. J., on June 18, 1957, and in St. Louis, Mo., on February 12, 1958.

The membership consists of 79 voting members, of whom 52 are classified as producers, 11 as consumers, and 16 as general interest members.

The committee suffered loss through resignation of R. R. Thurston, of The Texas Company; C. R. Eckert, of the Ruberoid Co.; and R. E. Green, of Thwing-Albert Instrument Co.

The committee has unanimously elected R. R. Thurston as an Honorary Member of the committee.

A new subcommittee has been formed to develop standards on bituminized fiber pipe.

The officers elected for the ensuing term of two years are as follows:

Chairman, H. R. Snoke.
Vice-Chairman, M. R. Beasley.
Secretary, G. W. Robbins.

All the standards and tentatives for which the committee is responsible have been reviewed and recommendations concerning them are set forth in this report.

REVISIONS OF TENTATIVES

The committee recommends revisions as indicated of the following tentatives and their continuation as tentative:

Tentative Methods of Sampling and Testing Felted and Woven Fabrics Saturated with Bituminous Substances for Use in Waterproofing and Roofing (D 146 – 56 T):[1]

Section 21.—Delete the third sentence and replace with the following: "Dry the ashing sample to constant weight at 221 ±5 F (105 ±3 C). Weigh to the nearest 0.01 g and ignite in a weighed porcelain or quartz dish or crucible until all carbon has been consumed."

Tentative Methods of Testing Asphalt-Base Emulsions for Use as Protective Coatings for Built-Up Roofs (D 1167 – 51 T):[2]

Section 14.—Revise the first sentence to read as follows: "Use a 6 by 6-in. test panel of brass approximately 0.03 in. thick with clean surfaces, free from oil, to which has been applied an emulsion coat as in Section 11, and which has been dried for 24 hours in a horizontal position in a forced draft air circulation oven at a temperature of 140 ± 5 F."

Tentative Specification for Asphalt Insulating Siding Surfaced with Mineral Granules (D 1226 – 52 T):[2]

Table I.—In column 3, decrease the minimum requirement for mineral matter passing a No. 100 Sieve from 40 to 35 per cent.

TENTATIVE REVISIONS OF STANDARDS

The committee recommends tentative

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* Sixty-first Annual Meeting of the Society. June 22–27, 1958.

[1] 1956 Supplement to Book of ASTM Standards, Part 3.

[2] 1955 Book of ASTM Standards, Part 3.

revisions as indicated of the following standards:

Standard Specifications for Asphalt Shingles Surfaced with Mineral Granules (D 225 - 54):[2]

Section 1(b).—In the tabulation of shingles, delete the references to types II and III in the line referring to hexagonal tab strips and corresponding values in inches.

Table I.—After "Weight of exposed area per 100 sq ft" revise to read as shown above:

	Type I		Type II		Type III	
	Max, lb	Min, lb	Max, lb	Min, lb	Max, lb	Min, lb
Weight of dry felt per 100 sq ft	...	10.0	...	10.0	...	10.0
Saturant of felt, per cent	...	165	...	165	...	165
Weight of weather side coating per 100 sq ft	...	18.5	...	23.5	...	26.0[b, c]
Weight of mineral matter per 100 sq ft passing No. 6 (3360-micron) and retained on No. 100 (149-micron) sieve	...	18.5	...	18.5	...	18.5[a]
Percentage by weight of mineral matter passing No. 100 (149-micron) sieve on basis of the sum of the bitumen in the coatings and mineral matter passing No. 100 (149-micron) sieve	55.0	...	55.0	...	55.0	...
Mineral matter (including surfacing and stabilizer), per cent of total weight of finished , shingle	...	...	...	...	...	35.0
Percentage of permissible ash in felt	10	...	10	...	10	...
Weight of coating material on unexposed weather side per 100 sq ft	...	...	...	...	...	10.0
Weight of mineral matter passing No. 6 (3360-micron) sieve and retained on No. 100 (149-micron) sieve on the unexposed weather side per 100 sq ft	...	...	...	...	...	14.0

[a] This shall be calculated as follows: $\frac{\text{net weight of packages inspected}}{\text{sq ft area per shingle} \times \text{number of shingles}} \times 100.$

[b] Thick-butt portion only.

[c] Exposed area only.

Standard Specification for Asphalt Roll Roofing Surfaced with Mineral Granules (D 249 - 56):[2]

Table I.—Add a new line as follows: "Maximum permissible ash in felt, per cent, ... 10."

Delete the line "Weight of saturant (soluble in CS_2) per 100 sq ft, min 16.0 lb, together with Footnote *a*, and substitute: "Saturation of felt, min, 160 per cent."

In the line "Weight of weather side coating per 100 sq ft" change the minimum from "14.8" to "18.5." Delete the maximum "32.4."

Change the value of "Percentage by weight of mineral matter passing No. 100 (149-micron) sieve on the basis of the sum of the bitumen in the coatings and the mineral matter passing No. 100 (149-micron) sieve" from "Maximum 50 per cent" to "Maximum 55 per cent."

Standard Method of Test for Coarse Particles in Mixtures of Asphalt and Mineral Matter (D 313 - 41):[2]

Section 6.—Add a new sentence to the definition of *B* following the equation, to read as follows: "To be obtained by ASTM Method D 4 - 52."

ADOPTION OF TENTATIVES AS STANDARD WITHOUT REVISION

The committee recommends that the following tentatives be approved for reference to letter ballot of the Society for adoption as standard without revision:

Tentative Specification for:

Wide Selvage Asphalt Roll Roofing Surfaced with Mineral Granules (**D 371 - 55 T**).

Tentative Methods of Testing:

Asphalt Emulsions for Use as Protective Coatings for Metal (**D 1010 - 55 T**), and
Asphalt Insulating Siding Surfaced with Mineral Granules (**D 1228 - 52 T**).

Tentative Method of Test for:

Staining Properties of Asphalts (Modified Pressure Method) (**D 1328 - 54 T**), and
Contact Compatibility Between Asphaltic Materials (Oliensis Test) (**D 1370 - 55 T**).

Methods D 1010 are under joint jurisdiction with Committee D-1 on Paint, Varnish, Lacquer, and Related Products, and accordingly this recommendation with respect to Methods D 1010 is subject to approval by Committee D-1.

ADOPTION OF TENTATIVE REVISIONS AS STANDARD

The committee recommends that the tentative revisions as indicated of the following standards be approved for reference to letter ballot of the Society for adoption as standard:

Standard Specifications for:

Asphalt Roll Roofing Surfaced with Powdered Talc or Mica (**D 224 - 55**) (Tentative Revision issued June, 1955),
Asphalt Shingles Surfaced with Mineral Granules (**D 225 - 54**) (Tentative Revision issued June, 1954, modified June, 1955), and
Asphalt Siding Surfaced with Mineral Granules (**D 699 - 54**) (Tentative Revision issued June, 1955).

WITHDRAWAL OF STANDARD

The committee recommends the withdrawal of Standard Specifications for Coal-Tar Pitch for Steep Built-Up Roofs (**D 654 - 49**). Materials meeting this specification are no longer available.

REAPPROVAL OF STANDARDS

The committee recommends the reapproval of the following standards which have stood for six or more years without revision:

Standard Specifications for:

Primer for Use with Asphalt in Dampproofing and Waterproofing (**D 41 - 41**),
Creosote for Priming Coat with Coal-Tar Pitch in Dampproofing and Waterproofing (**D 43 - 41**),
Bituminous Grout for Use in Waterproofing Above Ground Level (**D 170 - 41**),
Bituminous Grout for Use in Waterproofing Below Ground Level (**D 171 - 41**),
Woven Cotton Fabrics Saturated with Bituminous Substances for Use in Waterproofing (**D 173 - 44**),
Asphalt for Use in Constructing Built-Up Roof Coverings (**D 312 - 44**),
Asphalt for Dampproofing and Waterproofing (**D 449 - 49**),
Coal-Tar Pitch for Roofing, Dampproofing, and Waterproofing (**D 450 - 41**),
Asphalt Mastic for Use in Waterproofing (**D 491 - 41**),
Asphalt-Saturated and Coated Asbestos Felts for Use in Constructing Built-Up Roofs (**D 655 - 47**), and
Sieve Analysis of Granular Mineral Surfacing for Asphalt Roofing and Shingles (**D 1001 - 51**).

Standard Methods of Test for:

Steam Distillation of Bituminous Protective Coatings (**D 255 - 28**),
Sieve Analysis of Granular Mineral Surfacing for Asphalt Roofing and Shingles (**D 451 - 40**), and
Sieve Analysis of Nongranular Mineral Surfacing for Asphalt Roofing and Shingles (**D 452 - 40**).

Standard Methods of Testing:

Bituminous Mastics, Grouts, and Like Mixtures (**D 147 - 41**), and
Films Deposited from Bituminous Emulsions (**D 466 - 42**).

Standard Volume Correction Table for:

Tar and Coal-Tar Pitch (**D 633 - 44**).

Method D 633 is under joint jurisdiction with Committee D-4 on Road and Paving Materials, and this recommendation with respect to D 633 action is subject to approval by Committee D-4.

TENTATIVES CONTINUED WITHOUT REVISION

The committee recommends the continuation without revision of the following tentatives:

Tentative Specification for:

Asphalt-Base Emulsions for Use as Protective Coatings for Built-Up Roofs (**D 1227 - 52 T**).

Tentative Recommended Practice for:

Accelerated Weathering Test of Bituminous Materials (**D 529 - 39 T**).

EDITORIAL CHANGES

The committee recommends editorial changes as indicated in the following standards:

Standard Specification for Asphalt-Saturated Asbestos Felts for Use in Waterproofing and in Constructing Built-Up Roofs (D 250 - 56) (Tentative Revision issued June, 1957):[1]

Section 9.—In the tabulation of requirements, under 30-lb type, change the value of minimum weight of saturated felt per 100 sq ft (exclusive of wrapping and packing material) from 30 to 28.

Standard Method of Test for Steam Distillation of Bituminous Protective Coatings (D 255 - 28):[2]

Section 2.—Revise Paragraph (*f*) to read as follows: "(*f*) *Receiver.*—Graduated cylinders of uniform diameter having a capacity of 100 ml graduated in single ml."

Insert a new Paragraph (*g*) to read as follows: "(*g*) Separatory funnel.—A graduated separatory funnel having a capacity of not less than 500 ml."

Renumber the present Paragraph (*g*) as (*h*).

Delete the present Paragraph (*h*).

Section 3.—In line 12 change the words "separatory funnel" to read "graduated cylinder." In line 14 delete the comma after the word "condenser."

Replace the next to the last sentence with the following: "Combine the distillates in the separatory funnel and separate the recovered solvent from the water. Measure the volume of water-free solvent and retain it for further tests if required by the specifications."

Section 4.—Change the words "The results shall be reported" to "Report the results."

Figure 1.—Revise to show a 100-ml graduate instead of the separatory funnel.

Standard Specification for Asphalt-Saturated and Coated Asbestos Felts for Use in Constructing Built-Up Roofs (D 655 - 47) (Tentative Revision isued June, 1957):[2]

Section 9.—In the tabulation of requirements, under 50-lb type, change the minimum weight of finished product per 100 sq ft (exclusive of wrapping and packing material) from 50 to 48.

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[3]

This report has been submitted to letter ballot of the committee, which consists of 79 members; 59 members returned their ballots, of whom 57 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

H. R. SNOKE,
Chairman.

G. W. ROBBINS,
Secretary.

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[3] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

REPORT OF COMMITTEE D-9
ON
ELECTRICAL INSULATING MATERIALS*

Committee D-9 on Electrical Insulating Materials held three meetings during the year: in Atlantic City, N. J., during the Annual Meeting of the Society; in Virginia Beach, Va., on November 18, 19, and 20, 1957; and in Pittsburgh, Pa., on February 26, 27, and 28, 1958.

Present membership comprises 220, of whom 103 are classified as producers, 76 as consumers, and 41 as general interest members. Death of four members has been reported: V. B. Burgess, representative of Philadelphia Electric Co., J. H. Carll, representative of Asheville Mica Co., J. J. Chapman, representative of Johns Hopkins University, and J. W. Deaderick, representative of American Lava Corp.

During the year, new chairmen were appointed for Subcommittees V, VI, and XV. Subcommittee V has been completely reorganized, and an extensive review of specifications and methods in its jurisdiction is in progress.

Committee D-9 continues to take part in the work of other groups in the field of electrical insulation through representation on the International Electrotechnical Commission (IEC), the International Standards Organization (ISO), and the American Institute of Electrical Engineers (AIEE). Also, several ASTM tentatives and standards have been recommended for adoption by the American Standards Association (ASA) as American Standards. Additional discussion of these activities is contained in the Section on Activities of Subcommittees. Recommendations for adoption as American Standards will be found in the reports of Subcommittees I, III, VII, and XII.

A symposium sponsored by Subcommittee XI was held during the meeting in Virginia Beach on the subject of "Application of Statistics to D-9 Test Methods and Specifications." Other symposia are in prospect.

The officers elected for the ensuing term of two years are as follows:

Chairman, H. K. Graves.
Vice-Chairman, E. B. Snyder.
Recording Secretary, Thomas Hazen.
Membership Secretary, J. R. Taylor.

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1957 Annual Meeting, Committee D-9 presented the following recommendations to the Society through the Administrative Committee on Standards:

Tentative Specifications for:

General Purpose Grade GPO-1 Polyester Glass-Mat Sheet Laminate (**D 1532–58 T**),

Tentative Methods of Testing:

By Visual Examination of Used Electrical Oils in the Field (**D 1524–58 T**),
Approximate Acidity of Used Electrical Insulating Oil of Petroleum Origin (**D 1534–58 T**),
For Water in Insulating Liquids (Karl Fischer Method) (**D 1533-58 T**), and
For Dielectric Constant and Dissipation Factor of Polyethylene by the Liquid Displacement Procedure (**D 1531-58 T**).

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* **Sixty-first Annual Meeting of the Society, June 22–27, 1958.**

Revision of Tentative Specification for:

Absorbent Laminating Paper for Electrical Insulation (**D 1080–54 T**).

Revision of Tentative Methods of Test for:

High Voltage, Low Current Arc Resistance of Solid Electrical Insulating Materials (**D 495 – 56 T**),
Nonrigid Polyvinyl Tubing (**D 876 – 58 T**),
Pressure-Sensitive Adhesive Coated Tapes Used for Electrical Insulation (**D 1000 – 58 T**),
Hydrocarbon Waxes Used for Electrical Insulation (**D 1168 – 54 T**),
Corrosive Sulfur in Electrical Insulating Oils (**D 1275 – 53 T**), and
2,6 Ditertiary-Butyl Para-Cresol in New Electrical Insulating Oils (**D 1473 – 57 T**).

Revision of Tentative Recommended Practice for:

Purchase of Uninhibited Mineral Oil for Use in Transformers and Oil Circuit Breakers (**D 1040 – 54 T**).

Tentative Revision of Standard Method of:

Sampling Electrical Insulating Oils (**D 923 – 49**).

Revision of Standard and Reversion to Tentative of Specifications for:

Black Bias-Cut Varnished Cloth and Tape Used for Electrical Insulation (**D 373 – 55**).

Recommendations concerning D 1524 – 58 T, D 876 – 58 T, D 1000 – 58 T, and D 923 – 49 were accepted by the Standards Committee on February 28, 1958. The remaining recommendations were accepted on June 18, 1958, with the exception of D 373 – 55, which was accepted July 14, 1958.

RECOMMENDATIONS AFFECTING STANDARDS

The committee is presenting for publication three Suggested Methods; one new tentative; five adoptions of tentatives as standards without revision; one adoption of a tentative as standard with revisions; three adoptions of tentative revisions as standard; one revision of standard for immediate adoption; one withdrawal of standard; eight reapprovals of standards; and one editorial change. These recommendations are covered in the Appendix of this report.[1]

The committee has reviewed all other tentatives under its jurisdiction which have been published for two or more years and recommends that they be continued in their present status until prospective revisions are made.

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[2]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Insulating Varnishes, Paints, and Lacquers (W. E. Harvey, chairman).—The subcommittee has recommended to IEC Committee TC15 that, for testing varnish under IEC/15 Standard Methods of Test for Dielectric Strength of Electrical Insulating Materials at Commercial Power Frequencies (Revision 15 (Secretariat) 9) the top electrode shall be a 6-mm diameter cylinder with edges rounded to 0.075 mm radius, gasketed, and weighing 50 g; and that the bottom electrode shall be a metal plate.

For measuring viscosity of varnish, the subcommittee favors and is considering use of the Hoeppler viscosimeter in place of the MacMichael viscosimeter, now described in the Appendix to the Methods of Testing Varnishes Used for Electrical Insulation (D 115 – 55).

Studies are in progress or are proposed on resistance of insulating varnishes to Freon refrigerant gas; on mechanical measurement of gel time; on determination of cure time by helical coil; and on tests of varnish at high temperature.

The subcommittee recommends approval by ASA of Methods D 115 – 55 and D 1346 – 57 as American Standard.

Subcommittee II on Effects of Nuclear

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[1] See p. 352.

[2] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

and High Energy Radiation (D. S. Ballantine, chairman).—This joint D-9 and D-20 subcommittee has completed its first year of activity. Work of the subcommittee is organized into five sections. Section A on Nomenclature and Definitions has made a preliminary survey of preferences in terminology of units for expressing absorbed radiation dosage and is also compiling a glossary of nuclear terms. Sections B on Dosimetry, C on Exposure Methods, D on Education, and E on Research are gathering information in their fields. In particular, Section D is sponsoring a series of lectures on nuclear subjects, the first of which was presented by D. J. Metz and covered different types of radiation, modes of interaction, rates of absorption, and so on. Section E is contacting members of Committees D-9 and D-20 to determine cooperative interest in the use of equipment for fabrication of test specimens; the use of radiation facilities and physical testing equipment; and on questions of financing studies. There is sufficient interest to guarantee a small initial research effort, but this must be of general interest to other members of Committees D-9 and D-20.

At the present stage of nuclear development, Subcommittee II believes that the interests of the subcommittee and of its sponsoring committees would be best served by holding subcommittee meetings independently of the meetings of Committees D-9 and D-20. Therefore, the next meeting of the subcommittee will be on May 7, 8, and possibly 9, at the National Bureau of Standards in Washington, D. C.

Subcommittee III on Plates, Tubes, Rods and Molded Materials (E. A. Russell, chairman).—During the year the subcommittee recommended approval of a new Tentative Specification for General Purpose Grade GPO-1 Polyester Glass-Mat Sheet Laminate (D 1532 – 58 T) and revision of Methods D 876 – 54 T.

The subcommittee sponsors the recommendations concerning Methods D 229 – 49, D 617 – 44, D 634 – 44, D 651 – 48, D 668 – 52, and D 741 – 52, as set forth in the Appendix to this report. Methods D 634 – 44, D 668 – 52, and D 741 – 52 are recommended for approval as American Standards.

With regard to joint D-9 and D-20 jurisdictions, the subcommittee received approval from the D-9 Advisory Committee to retain Specification D 710 – 54 T under the sole jurisdiction of Committee D-9; to transfer to sole jurisdiction of Committee D-20 Specification D 700 – 55 T and Method D 785 – 51; and to continue joint D-9 and D-20 jurisdiction over Methods D 619 – 54 T, and Specifications D 709 – 55 T and D 1202 – 52 T.

Other tentatives sponsored by the subcommittee will be continued in effect except that Methods of Testing Molded Materials Used for Electrical Insulation (D 48 – 54 T) may be eliminated by direct references to test methods; no action was taken on Method of Measuring Shrinkage from Mold Dimensions of Molded Materials (D 551 – 41), because of possible replacement by the Method of Measuring Shrinkage from Mold Dimensions of Molded Plastics (D 955 – 51).

A task force set up at the June 1957 meeting has received from the Administrative Committee on Research authorization to organize a cooperative research project on measurement of insulation resistance of laminated thermosetting plastics, including metal surfaced plastics. The project will be a joint study of ASTM, NEMA, and possibly of other groups. An initial research appropriation of at least $1000 has been made available by the Administrative

Committee on Research, contingent on the extent of participation by other groups.

Other activity of the subcommittee includes evaluation of round-robin data on surface resistance of copper-clad XXXP and epoxy glass laminates to establish recommendations on electrodes and methods of measurement; methods of testing heat resistance and thermal stability of rigid electrical insulation; addition to Methods D 619 - 54 T of a section on resistance to the electric arc of unvulcanized fiber sheets; and consideration of applications of statistical methods to subcommittee test data.

Subcommittee IV on Liquid Insulation (F. M. Clark, chairman).—During the year the Administrative Committee on Standards accepted recommendations originating in the subcommittee on new Tentative Methods of Testing by Visual Examination of Used Electrical Oils in the Field (D 1524 - 58 T), for Approximate Acidity of Used Electrical Oils (D 1534 - 58 T), and for Water in Insulating Liquids (Karl Fischer Method) (D 1533 - 58 T); on revisions of the tentative Recommended Practice D 1040 - 54 T, and Tentative Methods D 1275 - 53 T and D 1473 - 57 T; on tentative revision of Standard Method D 923 - 49; on adoption of tentative as standard without revision Method D 1169 - 54 T; and on adoption of tentative as standard with revision Method D 117 - 54 T.

The subcommittee developed the suggested method for measuring oxidation stability of transformer oils[3] and a new tentative method for determining peroxide content of insulating oils.[4] The subcommittee has also sponsored recommendations for immediate adoption of tentative revisions as standard in Methods D 924 - 49, and for reapproval of Standard Methods D 831 - 48, D 877 - 49, and D 989 - 51. The subcommittee also authorized an editorial change in Methods D 1313 - 54.

The subcommittee continues liaison with other technical groups in the field of electrical insulation. Currently active are cooperative work with AIEE committees on joint AIEE-ASTM publication of a Guide for Maintenance of Insulating Oil and on development of test specifications for cable impregnating oils.

Subcommittee V on Ceramic Products (A. H. Scott, chairman).—During the year A. H. Scott was appointed chairman of the subcommittee. Under his direction a complete reorganization of activities has been undertaken. The subcommittee is sponsoring adoption of Tentative Methods D 1039 - 50 T as standard without revision; immediate adoption of revision of Specifications D 879 - 49 as standard and of a new standard based on ASA C-29 American Standard Test Methods for Electrical Power Insulators; and withdrawal of Standard Methods D 468 - 46 (contingent on acceptance of the ASA procedure) and D 550 - 44. The subcommittee has under consideration the revision and reversion to tentative of Specifications D 730 - 49.

Subcommittee VI on Solid Filling and Treating Compounds (Thomas Hazen, chairman).—Mr. W. R. Dohan was succeeded as chairman during the year by Mr. Hazen.

The subcommittee sponsored the revision of Methods D 1168 - 54 T. The revision provides for use in Methods D 1168 of Method D 1321 for measuring penetration of wax in place of the previously used Method D 5, and for necessary other changes.

Subcommittee VII on Insulating Fabrics (F. P. West, chairman).—During the year the Administrative Committee on Standards has approved the following recommendations which originated in the subcommittee: revision of Tentative Methods D 1000 – 58 T and revision and reversion to tentative of Specification D 373 – 55.

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[3] The suggested methods appear in a special reprint of the Annual Report of Committee D-9 on Electrical Insulating Materials.

[4] The new tentative appears in the 1958 Book of ASTM Standards, Part 9.

The subcommittee originated two Suggested Methods for Evaluation of Thermal Stability of Electrical Insulating Coated Fabrics by Curved Electrodes;[3] and recommended the adoption of Tentative Methods D 295 – 55 T as standard without revision.

The subcommittee also recommends the approval by ASA as American Standards of Methods D 295 – 55 T, D 902 – 56, and Specifications D 1459 – 57 T.

Other activities of the subcommittee have comprised a decision to modify Methods of Testing Varnished Glass Fabrics and Varnished Glass Fabric Tapes Used for Electrical Insulation (D 902 – 56) to make the method applicable to tests of polytetrafluoroethylene resin-coated glass fabrics; study of the effect of deliberate offset of the two electrodes in Systems 3 and 4 of IEC TC15/G1/UK/4; and a decision to submit to subcommittee letter ballot a proposed tentative specification for black or yellow straight cut oleoresin varnished cloth and varnished cloth tape used for electrical insulation, Issue No. 4, 10/7/57.

Subcommittee VIII on Insulating Papers (H. A. Anderson, chairman).—During the year the Administrative Committee on Standards approved the recommendation sponsored by the subcommittee on the revision of Tentative Specification D 1080 – 54 T.

The subcommittee also sponsored a recommendation that the method of test for water soluble chlorides in paper, an Appendix to Specifications D 1305 – 54 T, be transferred to Methods D 202 – 55 T as an Appendix.

Other work in progress in the subcommittee includes new round-robin tests to learn precision of methods of measuring chlorine content, conductivity, and pH; revision of the dry 60-cycle dissipation factor test; new round-robin tests on holes and felt hairs in capacitor tissue, and correlation with lacquering for metallizing paper; study of methods of measuring thickness of capacitor tissue when less than 1 mil; correlation of edge tear and folding endurance tests with taping performance of cable papers; study of oil-treated cable paper, preliminary to writing specifications in liaison with Subcommittee IV; and consideration of the applicability of statistical methods to subcommittee problems. A recommended practice for insulating paper is in preparation.

Subcommittee IX on Mica Products (K. G. Coutlee, chairman).—During the year, work has been in progress on development of the standard waviness test for mica, of interest to the U. S. General Services Administration; and on tests for bonded mica and mica paper. A proposed test for mica paper is being subjected to round-robin trial.

The subcommittee is cooperating with G. M. L. Sommerman, U. S. representative to IEC Working Group No. 1 of TC 15, by making electric strength measurements on mica. ASTM electrodes and United Kingdom unequal size electrodes are being used.

During the year, the subcommittee transferred to sole jurisdiction of Committee F-1 on Materials for Electron Tubes and Semiconductor Devices, the Methods for Measuring Mica Stampings Used in Electronic Devices and Incandescent Lamps (D 652 – 51).

Subcommittee X on Magnet Wire Insulation (K. N. Mathes, chairman).—The subcommittee, newly formed in the previous year, has under consideration a new specification for nylon insulated wire, including a solderability test; and wire for use at 130 C. Recommended for subcommittee letter ballot is a method for measuring frictional characteristics of enameled magnet wire used in field coils. Also in circulation are methods for measuring the mechanical properties, cut through, and abrasion; and for measuring exposure compatibility, in terms of solvent and refrigerant extractables, Refrigerant 22 blister test, and refrigerant aging test.

Subcommittee XI on Nomenclature, Significance, and Statistics (C. L. Craig, chairman).—The subcommittee has cooperated with other subcommittees on matters of mutual interest. It recommended to Committee D-9 in concurrence with Subcommittee III the revision of Methods D 876 – 54 T; and with Subcommittee VII the revision of Methods D 1000 – 57 T. To Subcommittee IV a recommendation was made for elimination from a revision of Method of Measuring DBPC in New Insulating Oils (D 1473 – 57 T) of a proposed definition statement, and a slight modification of a proposed significance statement was suggested.

During the year, the subcommittee sponsored a Symposium on Application of Statistics to D-9 Standards and obtained approval of the Advisory Committee to recommend publication as an ASTM Special Technical Publication.

A glossary of terms of interest to D-9 is in preparation, as is a compilation of a Statistical Practices Standard Acceptance Plan.

Subcommittee XII on Electrical Tests (N. W. Edgerton, chairman).—The subcommittee sponsored the new Tentative Method for Dielectric Constant and Dissipation Factor of Polyethylene under Liquid Displacement (D 1531 – 58 T); and revision of Tentative Method D 495 – 56 T.

The subcommittee sponsors adoption of Methods D 257 – 57 T as standard without revision. This method is also recommended for approval as an American Standard by ASA.

Subsequent to the 1957 Annual Meeting of the Society, three Suggested Methods sponsored by the subcommittee have been published in the 1957 Compilation of D-9 Specifications and Methods: Appendix IV, Method for Corona Measurement; Appendix V, Test for Dust and Fog Tracking Resistance of Electrical Insulating Materials; and Appendix VI, Liquid-Displacement Method for Dielectric Constant and Dissipation Factor of Polyethylene. As noted above, the procedure of Appendix VI has been advanced to tentative status.

The subcommittee continues work with other groups. IEC (15) Secretariat 15 on resistivity has been discussed, and recommendations were considered on use of formulas, instead of long descriptions, to specify conditioning procedures and testing conditions.

The subcommittee has cooperated with the AIEE Committee on Special Apparatus and Auxiliary Apparatus and with the Subcommittee on Corona. An AIEE Recommended Guide for Making Dielectric Measurements in the Field, now published on a proposed basis, is expected to be submitted for final approval.

Work is in progress in the sections on measurement of dielectric absorption and of very high resistances; on methods of designating conditioning and testing atmospheres; and on study of the electrostatic phenomena of insulating materials.

The subcommittee will sponsor a Symposium on Factors Affecting Dielectric Breakdown, to be held at the Fall 1958 Meeting of D-9, and a Symposium on Electrostatic Phenomena has been planned for the Spring 1959 Meeting.

Subcommittee XIV on Conditioning (G. M. Armstrong, chairman).—This joint D-9 and D-20 subcommittee sponsors adoption of tentative revisions of Methods D 618 – 54 as standard.

Further revision of D 618 – 54 is in progress which will bring it into essential agreement with recommendations of the Atmospheric Testing Coordinating Organization recently accepted by ISO/TC-61 and IEC/TC-15, and included in British and German standards.

Subcommittee XIV recommends that Committee D-9 join with Committee D-20 to transfer Specifications for Enclosures and Servicing Units for Tests Above and Below Room Temperature (D 1197 – 56) to Committee E-1.

This report has been submitted to letter ballot of the committee, which consists of 220 members; 96 members returned their ballots, of whom 80 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

H. K. GRAVES,
Chairman.

J. E. GIBBONS,
Secretary.

APPENDIX

RECOMMENDATIONS AFFECTING STANDARDS FOR ELECTRICAL INSULATING MATERIALS

In this Appendix are given the recommendations affecting certain standards covering electrical insulating materials referred to earlier in this report. The standards and tentatives, excepting those adopted later, appear in the 1955 Book of Standards, Part 6. Subsequent adoptions are to be found in the Committee D-9 compilations of specifications and methods of testing in 1956 and 1957.

SUGGESTED METHODS TO BE PUBLISHED AS INFORMATION

The committee recommends for publication as information only the following three suggested methods as appended hereto:[1]

Suggested Method of Test for:

Evaluation of Thermal Stability of Electrical Insulating Coated Fabrics by Curved Electrodes (Dielectric Prooftest Method),

Evaluation of Thermal Stability of Electrical Insulating Coated Fabrics by Curved Electrodes (Dielectric Breakdown Method), and

Oxidation Characteristics of Mineral Transformer Oil.

The committee believes there is a need for tests of this type and that publication as suggested methods makes it possible to get early test data, comments, and criticism, from which a decision to publish as a tentative method can be made at an earlier date than would otherwise be possible.

NEW TENTATIVE

The committee recommends for publication as tentative the Method of Test for Peroxide Number of Mineral Insulating Oils, as appended hereto.[2]

This method provides a definite procedure, not heretofore available, for measurement of peroxides which are sometimes found in oxidized mineral oils.

ADOPTION OF TENTATIVES AS STANDARD WITHOUT REVISION

The committee recommends that the following tentatives be approved for reference to letter ballot of the Society for adoption as standard without revision:

Tentative Methods of Test for:

Electrical Resistance of Insulating Materials (**D 257 – 57 T**),

Insulation Resistivity of Electrical Insulating Oils of Petroleum Origin (**D 1169 – 54 T**), and

Corrosive Sulfur in Electrical Insulating Oils (**D 1275 – 58 T**).

Tentative Methods of Testing:

Varnished Cotton Fabrics and Varnished Cotton Fabric Tapes Used for Electrical Insulation (**D 295 – 55 T**), and

Glass-Bonded Mica Used as Electrical Insulation (**D 1039 – 50 T**).

ADOPTION OF TENTATIVE AS STANDARD WITH REVISION

The committee recommends that the Tentative Methods of Testing Electrical Insulating Oils (**D 117 – 54 T**)[3] be approved for reference to letter ballot of the Society for adoption as standard with revisions as indicated.

Table I.—Delete the entire line entitled "Steam Emulsion."

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[1] The suggested methods appear in a special reprint of the Annual Report of Committee D-9 on Electrical Insulating Materials.

[2] The new tentative appears in the 1958 Book of ASTM Standards, Part 9.

[3] 1955 Book of ASTM Standards, Part 6.

Center Heading.—Delete the center heading "Steam Emulsion."

Sections 44, 45, and 46.—Delete these sections, renumbering the subsequent sections accordingly.

These deletions and renumbering of sections are needed to take account of the withdrawal from the Book of Standards of the Steam Emulsion Test (D 157).

ADOPTION OF TENTATIVE REVISIONS AS STANDARD

The committee recommends that the tentative revisions as indicated of the following standards be approved for reference to letter ballot of the Society for adoption as standard:

Methods of Testing Sheet and Plate Materials Used for Electrical Insulation (D 229 – 49):[3]

Incorporate in the text of D 229 – 49 the various revisions now appended to the method as Editorial Notes, omitting the tentative revision of Section 27, issued in June 1951. Renumber and retitle sections as needed.

Methods of Conditioning Plastics and Electrical Insulating Materials for Testing (D 618 – 54):[3]

Incorporate in the text of D 618 – 54 the tentative revisions dated June 1956, and now appended as an Editorial Note. Renumber sections as needed.

Standard Method of Test for Power Factor and Dielectric Constant of Electrical Insulating Oils of Petroleum Origin (D 924 – 49):[3]

Incorporate in the text of Method D 924 – 49 the tentative revisions now appended as an Editorial Note.

REVISION OF STANDARD, IMMEDIATE ADOPTION

The committee recommends for immediate adoption revisions as follows of the Standard Specification for Communication and Signal Pin-Type Lime-Glass Insulation (**D 879 – 49**),[3] and accordingly asks for the necessary nine-tenths affirmative vote at the Annual Meeting in order that the revisions may be referred to letter ballot of the Society.

Section 3.—Replace with the following:

3. (*a*) The thermal shock test shall consist of alternate immersions of the insulators in hot and cold water baths. The transfer may be by manual or machine method.

(*b*) *Testing Arrangement.*—The insulators shall be mounted in a horizontal position on a rack or similar support so arranged that the insulators are not in contact with each other and that the air shall not be trapped during immersion. The rack design shall permit free circulation of water. Test specimens shall be at least 2 in. from the walls of the tank. At least four insulators of each type shall be tested at one time.

(*c*) *Water Baths.*—Each bath shall have a weight of water at least 10 times the weight of the insulators immersed. Either natural or forced circulation may be used to maintain the temperature of all parts of the bath within the specified values stated below. The recorded temperature shall be measured at least 4 in. from the heating or cooling elements.

(*d*) *Method of Making Test.*—The insulators shall first be immersed in a hot water bath for 10 min, then they shall be withdrawn and immersed in a cold water bath for 10 min. For this hot to cold test, the temperature of the hot water bath shall be between 120 and 140 F (49 and 60 C) and the temperature difference between the hot and cold baths at the time of immersion shall be 70 ± 2 F (39 ± 1 C).

For the cold to hot test, the time of immersion shall be the same as in the hot to cold test, but the temperature of hot water bath shall be between 150 and 170 F (66 to 77 C). The temperature difference between the cold and hot baths at the time of immersion shall be 100 ± 2 F (56 ± 1 C). The transfer time between the two baths shall not exceed 15 sec. After the hot to cold and again after the cold to hot immersions, the insulators shall be checked for cracks, chips, and spalls. Tested insulators shall be discarded.

Section 5(b).—Revise to read as follows:

(*b*) *Screw Threads.*—The insulators shall have the standard screw threads to fit the wooden-pin gage or the CSC gage, as applicable. The dimensions and construction of these gages shall be as described in Figs. 2 and 3, respectively. The thread shall be smooth, of uniform pitch, and well-centered in the insulator.

New Figures.—Add two new figures, Fig. 2, Gage for Wooden-Pin Insulators, and Fig. 3, Gage for Carrier-Current Insulators (CSC). The new figures shall be the same as Figs. 2 and 3 of Methods D 468 – 46.

Section 11.—Revise to read as follows:

> 11. The specimen insulators shall be subjected to the stipulated thermal shock test (manual method or machine method) as described in Section 3.

Section 18.—Revise the first sentence to read as follows:

> 18. The other half of the sample lot shall be tested, for thermal shock in accordance with Section 3.

WITHDRAWAL OF STANDARDS

The committee recommends the withdrawal of the Standard Methods of Testing Glass Spool Insulators (**D 550 – 44**).[3]

REAPPROVAL OF STANDARDS

The committee recommends the reapproval of the following standards which have stood for six or more years without revision:

Methods of Test for:

Punching Quality of Phenolic Laminated Sheets (**D 617 – 44**),

Product Uniformity of Phenolic Laminated Sheets (**D 634 – 44**),

Tensile Strength of Molded Electrical Insulating Materials (**D 651 – 48**),

Gas Content of Insulating Oils (**D 831 – 48**),

Dielectric Strength of Insulating Oil of Petroleum Origin (**D 877 – 49**), and

Free Sulfur in Electrical Insulating Oils (**D 989 – 51**).

Methods of Measuring:

Dimensions of Rigid Tubes Used for Electrical Insulation (**D 668 – 52**), and

Dimensions of Rigid Rods Used for Electrical Insulation (**D 741 – 52**).

EDITORIAL CHANGE

The committee recommends an editorial change as follows in the Method of Test for Sludge Formation in Mineral Transformer Oil by High-Pressure Oxidation Bomb (**D 1313 – 54**):[3]

Section 1(b).—Delete the last sentence which reads: "No data are available that correlate the results obtained with the commercial use of transformer oil."

REPORT OF COMMITTEE D-10
ON
SHIPPING CONTAINERS*

The last report of Committee D-10 on Shipping Containers was presented to the Society at the 1956 Annual Meeting. Since that time, the committee has held four meetings: on October 23 and 24, 1956, in St. Louis, Mo.; on April 9 and 10, 1957, in Chicago, Ill.; on October 30 and 31, 1957, in Atlantic City, N. J.; and on May 26 and 27, 1958 in Stamford, Conn.

Committee D-10 consists of 103 members, 34 of whom are classified as producers, 41 as consumers, and 28 as general interest members.

The officers elected for the ensuing term of two years are as follows:

Chairman, J. G. Turk.
Vice-Chairman, K. W. Kruger.
Secretary, R. F. Uncles.
Advisory Committee, R. E. Jones and A. V. Grundy.

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1957 Annual Meeting, Committee D-10 presented to the Society through the Administrative Committee on Standards the recommendation that the Standard Method of Drop Test for Shipping Containers (D 775 – 47) be revised and reverted to tentative status. This recommendation was approved by the Standards Committee on December 13, 1957, and the revised method appears in the 1957 Supplement to Book of ASTM Standards, Part 7.

TENTATIVES CONTINUED WITHOUT REVISION

The committee recommends the continuation without revision of the following tentatives:

Tentative Methods of:

Vibration Test for Shipping Containers (**D 999 – 48 T**),
Testing Pallets (**D 1185 – 51 T**),
Test for Water Vapor Permeability of Packages by Cycle Method (**D 1251 – 53 T**),
Test for Water Vapor Permeability of Shipping Containers by Cycle Method (**D 1276 – 53 T**), and
Test for Package Cushioning Material (**D 1372 – 55 T**).

Tentative Definitions of:

Terms Relating to Shipping Containers (**D 996–56 T**).

Adoption as standard is not warranted for Methods D 1185, D 1251, D 1276, and D 1372 since their adequacy has not been established by field application. Methods D 1251 and D 1276 are scheduled for review and possible revision by Subcommittee III. Methods D 996 and D 999 are under investigation by the committee.

REAPPROVAL OF STANDARDS

The committee recommends the reapproval of the following standards which have stood without revision for six or more years:

Standard Method of:

Compression Test for Shipping Containers (**D 642 – 47**),

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* Sixty-first Annual Meeting of the Society, June 22 to 27, 1958.

Incline Impact Test for Shipping Containers (**D 880 - 50**),
Drop Test for Bags (**D 959 - 50**),
Drop Test for Cylindrical Shipping Containers (**D 997 - 50**),
Test for Water Vapor Permeability of Packages (**D 895 - 51**),
Test for Water Resistance of Containers by Spray Method (**D 951 - 51**),
Test for Penetration of Liquids into Submerged Containers (**D 998 - 51**), and
Test for Water Vapor Permeability of Shipping Containers (**D 1008 - 51**).

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[1]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Definition of Terms (C. S. Macnair, chairman) has continued its resolution of groups of terms to be offered for inclusion in Tentative Definitions of Terms Relating to Shipping Containers (D 996 - 56 T).

Subcommittee II on Methods of Testing (J. G. Turk, chairman),

Subcommittee IV on Performance Standards (C. R. Gustafson, chairman), and

Subcommittee V on Correlation of Tests (R. C. McKee, chairman).—These three subcommittees continue to function as a joint committee consisting of task groups, each investigating established test methods whose reproducibility has been questioned. A revision of the Test for Shipping Containers in Revolving Hexagonal Drum (D 782) will be presented shortly for committee approval. The Vibration Test Task Group and Stacking Test Task Group are progressing in their assignments. A new task group has been organized to study the Incline Impact Test for Shipping Containers.

Subcommittee III on Water Vapor Resistance (H. A. Bergstrom, chairman) has completed a revision of the Test for Water Vapor Permeability of Shipping Containers (D 1008) and will present the revision to Committee D-10 for approval. Other water vapor permeability test methods will be reviewed.

Subcommittee VI on Interior Packing (K. Q. Kellicutt, chairman) has progressed in its investigations on resiliency, breakdown and dusting, dunnage, and reaction on finishes. The Dynamic Testing group will present a tentative procedure at an early date.

This report has been submitted to letter ballot of the committee, which consists of 103 members; 100 members returned ballots, of whom 86 voted affirmatively and 1 negatively.

Respectfully submitted on behalf of the committee,

G. E. FALKENAU,
Chairman.

R. F. UNCLES,
Secretary.

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[1] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

REPORT OF COMMITTEE D-11
ON
RUBBER AND RUBBER-LIKE MATERIALS*

Committee D-11 on Rubber and Rubber-Like Materials and its subcommittees has continued its high level of activity during the year. Two meetings have been held: in Atlantic City, N. J., on June 19, 1957 and in St. Louis, Mo., on February 12, 1958. These meetings resulted in a number of new tentatives and revisions of tentatives.

The committee sponsored a Symposium on Effect of Ozone on Rubber at St. Louis during ASTM Committee Week. This symposium will be published as a separate publication *ASTM STP No. 229.*

The Advisory Committee, upon reviewing the publication of the D-11 Compilation of Standards, is recommending that after the 1958 revised compilation is published, a new compilation be issued every second year rather than yearly. In the intervening years all revisions and new tentatives will be made available through the Annual Report.

The committee has continued to study the problem of the definitions for "rubber" and "rubber-like" in order to try to clarify the division of responsibility between Committee D-11 and Committee D-20 on Plastics. The results of this study are being proposed as part of the Tentative Definitions of Terms Relating to Rubber and Rubber-Like Materials.

Mr. W. A. Frye of the Inland Manufacturing Division of General Motors Corp. has been appointed chairman of Subcommittee XXVII on Tests of Resilience, replacing E. G. Kimmich who found it necessary to resign. Mr. G. W. Painter of the Lord Manufacturing Co. is the new secretary of this subcommittee.

In view of the meeting of the ISO/TC 45 committee in the United States in October 1959 it is not feasible for Committee D-11 to participate in the 1959 Pacific Area National Meeting in San Francisco.

Plans for the meeting of the ISO Technical Committee 45 are progressing. The meeting will be held in New York City on October 26 to 31, 1959.

Plans for the International Rubber Conference in which Committee D-11 is cooperating are also progressing. The meeting will be held in Washington, D. C., the week of November 8, 1959.

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1957 Annual Meeting, Committee D-11 presented to the Society through the Administrative Committee on Standards the following recommendations:

New Tentative Methods for:

Testing Melting Range of Chemicals (**D 1519 – 58 T**),

Synthetic Rubber Heat and Moisture Resisting Insulation for Wire and Cable (**D 1520 – 58 T**),

Synthetic Rubber, Performance Type, Moisture Resisting, Insulation for Wire and Cable (**D 1521 – 58 T**).

Revision of Tentative Methods of:

Testing Rubber and Thermoplastic Insulated Wire and Cable (**D 470 – 56 T**),

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* Sixty-first Annual Meeting of the Society, June 22–27, 1958.

Test for Indentation of Rubber by Means of the Durometer (**D 676 – 55 T**),
Measuring Low Temperature Stiffening of Rubber and Rubber-Like Materials by the Gehman Torsional Apparatus (**D 1053 – 54 T**),
Test for Evaluating Low Temperature Characteristics of Rubber and Rubber-Like Materials by a Temperature Retraction Procedure (**D 1329 – 54 T**), and
Chemical Analysis of Synthetic Elastomers (**D 1416 – 56 T**).

Revision of Tentative Specifications and Methods of Test for:

Latex Foam Rubbers (**D 1055 – 56 T**), and
Sponge and Expanded Cellular Rubber Products (**D 1056 – 56 T**).

Revision of Tentative Specifications for:

Elastomer Compounds for Automotive Applications (**D 735 – 57 T**), and
Construction of Rubber Insulated Wire and Cable (**D 1350 – 56 T**).

Revision of Recommended Practices for:

Standard Test Temperature for Rubber and Rubber-Like Materials (**D 1349 – 54 T**),
Nomenclature for Synthetic Elastomers and Latices (**D 1418 – 56 T**),
Description of Types of Styrene-Butadiene Rubbers (SBR) (**D 1419 – 56 T**), and
Description of Types of Styrene-Butadiene (SBR) and Butadiene (BR) Latices (**D 1420 – 56 T**).

These recommendations were accepted on February 28, 1958, with the exception of Methods D 1416 and D 1418. The latter were accepted on March 31, 1958. They appear in the 1958 Compilation of ASTM Standards on Rubber and Rubber-Like Materials.

On April 16, 1958 the following recommendations were accepted by the Administrative Committee on Standards and they appear in the Compilation of ASTM Standards on Rubber and Rubber-like Materials:

New Tentative Methods for:

Testing Carbon Blacks in Rubber (**D 1522 – 58 T**).

Revision of Tentative Specifications for:

Non-Metallic Gasket Materials for General Automotive and Aeronautical Purposes (**D 1170 – 54 T**).

Revision of Tentative Recommended Practices for:

Description of Types of Styrene-Butadiene Rubbers (SBR) (**D 1419 – 56 T**), and
Description of Types of Styrene-Butadiene Rubber (SBR) and Butadiene Rubber (BR) Latices (**D 1420 – 56 T**).

On April 25, 1958, the following recommendations were accepted by the Administrative Committee on Standards, and appear in the Compilation of ASTM Standards on Rubber and Rubber-like Materials:

New Tentative Specifications for:

Synthetic Rubber Insulation for Wire and Cable for 90 C Operation (**D 1523 – 58 T**).

Revision of Tentative Specifications for:

Natural Rubber AO Insulation for Wire and Cable (**D 27 – 54 T**),
Natural Rubber Performance Insulation for Wire and Cable (**D 353 – 54 T**),
Natural Rubber Heat Resisting Insulation for Wire and Cable (**D 469 – 54 T**),
Synthetic Rubber Heat Resisting Insulation for Wire and Cable (**D 754 – 54 T**),
Synthetic Rubber Performance Insulation for Wire and Cable (**D 755 – 54 T**), and
Construction of Rubber Insulated Wire and Cable (**D 1350 – 57 T**).

NEW TENTATIVES

The committee recommends for publication as tentative the following as appended hereto:[1]

Specifications and Methods of Test for Flexible Foams Made from Polymers or Copolymers of Vinyl Chloride

These methods were prepared by the coordinating committee of SPI and have been reviewed by Subcommittee XXII, the SAE-ASTM Technical Committee on Automotive Rubber, and the RMA Foam Latex Technical Committee. They apply to cellular products containing interconnecting cells made of a base material known as poly(vinyl chloride) or copolymers thereof.

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[1] The new and revised tentatives appear in the 1958 Book of ASTM Standards, Part 9.

Specifications and Methods of Test for Flexible Urethane Foam

These methods were prepared by the Coordinating Committee of the SPI, RMA Foam Latex Technical Committee, Subcommittee XXII of Committee D-11 and the SAE-ASTM Technical Committee on Automotive Rubber.

Definitions of Terms Relating to Rubber and Rubber-Like Materials

In order to define the terms "rubber" and "rubber-like" as used in the title and scope of Committee D-11, a task group is recommending revisions in the *ASTM Special Technical Publication No. 184*, "Glossary of Terms Relating to Rubber and Rubber-Like Materials." Subcommittee VIII reviewed these proposed revisions and has recommended that they be adopted not only for the Glossary but also as Tentative Definitions as appended hereto.[1]

Subcommittee VIII also has reviewed the ISO/TC 45 definitions of a number of terms. It is recommended that they also be approved as revisions in the Glossary and also for inclusion in the above recommended practice for nomenclature.

REVISIONS OF TENTATIVES

The committee recommends revisions as indicated of the following tentatives and their continuation as tentative:

Tentative Methods of Sample Preparation for Physical Testing of Rubber Products (D 15 - 57 T):[2]

Table II.—Add the following new SBR polymers to Table II as indicated:

SBR 1551: Add to column under 2B and remove the reference to 1551 from the Note.
SBR 1061: Add to column under 1B.
SBR 1605: Add to column under 6B.
SBR 1773: Add to column under 2B.
SBR 1778: Add to column under 2B.

Tentative Methods of Testing Rubber-Coated Fabrics (D 751 - 57 T):[2]

Section 14 (b).—Change to read as follows:

(*b*) *Diaphragm Bursting Tester.*—Requirements for a diaphragm bursting tester are outlined below; however, any machine that operates on the same principle and has coaxial apertures of 1.23 ± 0.02 in. in diameter in the clamping surfaces is a valid machine for this test.

(*1*) The testing machine to be used may be either mechanically or manually operated. It shall permit the clamping of the material to be tested between two circular clamps not less than 3 in. in diameter having coaxial apertures of their centers 1.23 ± 0.02 in. in diameter. The surfaces of the clamps between which the specimen is to be placed shall be concentrically grooved. The grooves shall be spaced not less than $\frac{1}{32}$ in. apart and of a depth not less than 0.006 in. The grooves shall not start closer than 0.125 in. from the edge of the aperture. The surfaces of the clamps shall be metallic and any edge which might cause a cutting action shall be rounded to a radius of not over $\frac{1}{64}$ in. The lower clamp shall be integral with the chamber in which a screw shall operate to force a liquid pressure medium at a uniform rate of 6.00 ± 0.25 cu in. per min against a rubber diaphragm fitted to expand through the aperture exerting its force against the coated fabric set between the two clamps. (In the manually operated machine, this shall correspond to approximately 120 rpm of a hand wheel turning the displacement screw.)

(*2*) In the mechanically operated machine, means shall be provided for stopping, at the instant of the rupture of the specimen, any further application of the loading pressure, and for holding unchanged the contents of the pressure chamber until the gross bursting pressure and tare diaphragm pressure indicated on the gage have been recorded. The machine shall be fitted with a Bourdon tube gage maximum hand type having a dial 4.5 in. in diameter, the scale divided to read in 0.5-lb units, with a range of from 0 to 100 lb. Accuracy shall be within plus or minus 0.5 lb. When the gage is calibrated, it shall be mounted in the same relative position as on the bursting tester. If calibrated with a dead-weight tester, the weights shall be spun slowly to insure free action of the piston.

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[2] 1957 Supplement to Book of ASTM Standards, Part 6.

NOTE.—A higher capacity gage may be used provided it meets the accuracy requirements.

(*3*) Provision shall be made for applying to the specimen, before clamping, a slight initial tension which shall be uniform in all directions.

Section 15.—In the first line, change "ten test specimens" to read "ten specimens."

Section 16 (*b*).—Change to read as follows:

(*b*) When the diaphragm tester is used, the specimen shall be mounted between the ring clamp and the base surface with a uniform tension. The pressure shall be applied at a constant rate as prescribed in Section 14 (*b*) Item (*1*) until rupture occurs. The pounds pressure shall be recorded and then the upper clamp shall be completely released, allowing the tare of the diaphragm to register on the pressure gage. The bursting strength of the material shall be reported as the gross pounds per square inch minus the tare of the diaphragm in pounds per square inch.

Section 17.—In the second line change the words "the coated fabric" to read "the material."

Tentative Specifications and Methods of Test for Latex Foam Rubbers (D 1055 – 58 T):[3]

Section 21.—In the first sentence after the words "connected to a load-measuring device" insert the words, "by means of a ball-and-socket joint." Also in the first sentence, change the wording from "can be deflected at a specified rate" to read "can be deflected at a rate of 0.5 to 25.0 in. per min." At the end of the Section add a Note to read: "NOTE.—When testing products with parallel top and bottom surfaces the ball-and-socket joint is not required."

Section 22.—Change the last sentence from "The full thickness of the product shall be used" to read, "The thickness shall be no less than $\frac{3}{4}$ in."

Section 23.—At the end of Paragraph (*a*) add the words, "except where the contour makes this impractical."

Change the second sentence of Paragraph (*b*) to read, "The *specimen* shall then be compressed 25 per cent of this original height and the final load in pounds *including the one pound preload* observed *approximately 5 sec after the foot has come to rest.*"

In the second sentence of Paragraph (*c*), change the word "product" to "specimen."

Tentative Method of Test for Evaluating Pressure Sealing Properties of Rubber and Rubber-Like Materials (D 1081 – 49 T):[4]

New Footnote.—Add a new Footnote 3 referenced to Section 2, Apparatus, to show a source of a seal aging jig, as follows: "[3] A suitable seal aging jig meeting these requirements can be obtained from the Precision Scientific Co., 3737 W. Courtland Ave., Chicago 47, Ill., Catalogue No. 15140."

Tentative Methods of Test for Harmful Dirt in Crude Natural Rubber (D 1278 – 53 T):[4]

Revise as appended hereto.[1]

These revised methods are based on the recommended procedures of the ISO/TC 45 Committee on Rubber. They include sampling of the rubber and chemical analysis of the sample for volatile matter, dirt, ash, copper, manganese, iron, acetone extract, and rubber hydrocarbon.

Tentative Methods for Chemical Analysis of Synthetic Elastomers (Solid Butadiene-Styrene Copolymers) (D 1416 – 58 T):[3]

New Method.—Add the following

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[3] Compilation of ASTM Standards on Rubber and Rubber-Like Materials, May, 1958.

[4] 1955 Book of ASTM Standards, Part 6.

Method for Determination of Mixed Alkylated Phenols (MAP) Content as Sections 62 through 69:

DETERMINATION OF MIXED ALKYLATED PHENOLS (MAP) CONTENT

Scope and Application

62. (*a*) This method is an ultraviolet absorption technique which is applicable to rubbers made with carbamate shortstop but not to oil-master-batch rubbers. Interference from carbamate shortstop is reduced by using an ETA extract of the rubber rather than a complete solution of the rubber. The high absorbance of oil, at the wavelengths used in this method, masks the measurement of the mixed alkylated phenols absorption.

(*b*) The difference between the basic and neutral absorbance at 301 mμ is proportional to the concentration of the mixed alkylated phenols. The neutral absorbance measurement is a background correction for substances other than the active ingredients.

Summary of Method

63. Two aliquots of the ETA extract are taken; one is left neutral, the other is made 0.1 *N* in base with alcoholic potassium hydroxide. The absorbance of both solutions is measured at 301 mμ, compared to corresponding reference solutions. The difference between the basic and neutral absorptions is proportional to the concentration of the mixed alkylated phenols.

Apparatus

64. (*a*) *Ultraviolet Photoelectric Spectrophotometer.*[7]

(*b*) *Matched Quartz Absorption Cells*, 1-cm.

(*c*) *Pipets*, 5-ml.

(*d*) *Volumetric Flasks*, 50-ml.

(*e*) *Analytical Balance.*

Reagents

65. (*a*) *Ethanol-Toluene-Azeotrope* (*ETA*).—Refer to Section 27 (*a*).

(*b*) *Ethanol*, anhydrous, Formula 2-B.

(*c*) *Potassium Hydroxide* (*0.5 N*) in alcohol. Grind 14 g of potassium hydroxide with successive 50-ml portions of anhydrous ethanol until dissolution is complete. Dilute to 400 ml with anhydrous ethanol. Filter immediately into a dark bottle and keep tightly closed. Let this solution stand overnight before using. When it becomes noticeably discolored, it should be discarded. Storing in a nitrogen atmosphere will preserve the solution considerably.

Preparation of Sample

66. Prepare the sample in accordance with Section 33.

Determination of Absorptivity

67. The spectrophotometric measurements shall be made the same day as the extraction is done.

(*a*) Make a solution of 0.300 g of mixed alkylated phenols (MAP) in 1 liter of anhydrous ethanol (ethanol MAP solution).

(*b*) Prepare the basic and neutral solutions in two separate 50-ml volumetric flasks as follows:

Basic Solution

5 ml ethanol-MAP solution
5 ml ETA solvent
10 ml 0.5 *N* potassium hydroxide
35 ml ethanol

Neutral Solution

5 ml ethanol-MAP solution
5 ml ETA solvent
40 ml ethanol

(*c*) Prepare the corresponding reference solutions as follows:

Basic Reference

5 ml ETA solvent
10 ml 0.5 *N* potassium hydroxide
40 ml ethanol

Neutral Reference

5 ml ETA solvent
45 ml ethanol

(*d*) Measure the absorbance of each solution at 301 mμ, using the corresponding reference solution in the spectrophotometer reference cell in each case. Call the observed absorbance values A_{basic} and A_{neutral}, respectively. Calculate the difference in absorptivity ($a_{\text{basic}} - a_{\text{neutral}}$) as follows:

$$(a_{\text{basic}} - a_{\text{neutral}}) = \frac{(A_{\text{basic}} - A_{\text{neutral}})}{(0.030)}$$

Where:

A_{basic} = measured absorptivity of basic solution,
A_{neutral} = measured absorptivity of neutral solution,
a_{basic} = measured absorption of basic reference solution, and
a_{neutral} = measured absorption of neutral reference solution.

Procedure

68. (*a*) Extract 6 g of rubber with two 100-ml portions of ETA for 1 hr each in accordance with

Section 34 (*a*) and (*b*), including the final dilution to 250 ml.

(*b*) Prepare the basic and neutral solutions in two separate 50-ml volumetric flasks as follows:

Basic Solution

5 ml ETA extract
10 ml 0.5 *N* potassium hydroxide
40 ml ethanol

Neutral Solution

5 ml ETA extract
45 ml ethanol

(*c*) Prepare the corresponding reference solutions as follows:

Basic Reference

5 ml ETA solvent
10 ml 0.5 *N* potassium hydroxide
40 ml ethanol

Neutral Reference

5 ml ETA solvent
45 ml ethanol

(*d*) Measure the absorbance of each solution at 301 mμ using the corresponding reference solution in the spectrophotometer reference cell in each case. Call the observed absorbance values A_{basic} and A_{neutral}, respectively.

Calculation

69. Calculate the percentage of mixed alkylated phenols (MAP) content as follows:

$$\text{MAP, per cent} = \frac{(A_{\text{basic}} - A_{\text{neutral}})\ 100}{(2.4)\ (a_{\text{basic}} - a_{\text{neutral}})}$$

ADOPTION OF TENTATIVES AS STANDARD WITHOUT REVISION

The committee recommends that the following tentatives be approved for reference to letter ballot of the Society for adoption as standard without revision:

Tentative Specifications for:

Natural Rubber AO Insulation for Wire and Cable (**D 27 - 58 T**),
Natural Rubber Performance Insulation for Wire and Cable (**D 353 - 58 T**),
Natural Rubber Heat-Resisting Insulation for Wire and Cable (**D 469 - 58 T**),
Natural Rubber Sheath for Wire and Cable (**D 532 - 54 T**),
Ozone-Resisting Insulation for Wire and Cable (**D 574 - 54 T**),
Synthetic Rubber Insulation for Wire and Cable, 75 C Operation (**D 754 - 58 T**),
Synthetic Rubber Insulation for Wire and Cable, 60 C Operation (**D 755 - 58 T**), and
GR-S Synthetic Rubber Sheath for Wire and Cable (**D 866 - 54 T**).

Tentative Methods of:

Test for Hardness of Rubber (**D 314 - 52 T**),
Test for Adhesion of Vulcanized Rubber to Metal (**D 429 - 56 T**),
Test for Compression Fatigue of Vulcanized Rubber (**D 623 - 52 T**), and
Testing Adhesives for Brake Lining and Other Friction Materials (**D 1205 - 53 T**).

REVISIONS OF STANDARD, IMMEDIATE ADOPTION

The committee recommends for immediate adoption revisions as follows of the Standard Method of Test for Young's Modulus in Flexure of Natural and Synthetic Elastomers at Normal and Subnormal Temperatures (**D 797 - 46**):[4]

Section 4.—In the fifth and seventh lines of Paragraph (*a*) change "70 F" to read "73 F."

In the last line of Paragraph (*c*) change "70 F" to read "73 F."

In the third line of Paragraph (*d*) change "−70 F" to read "−67 F." In the seventh line change "+70 F" to read "+73 F."

TENTATIVE CONTINUED WITHOUT REVISION

The committee recommends the continuation without revision of the remaining tentatives under its jurisdiction. All tentatives have been reviewed by the subcommittees and many are under consideration for revision.

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[5]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee V on Insulated Wire and Cable (J. T. Blake, chairman) is continuing to work on specifications for silicone rubber insulation. It is also concerned with the standardization of ovens for heat aging. It is cooperating with Subcommittee XV and Committee D-20 on this problem.

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[5] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

Subcommittee VI on Packings (R. F. Anderson, chairman) is making progress in its consideration of tests for compressibility and recovery of gasket materials. It is in close touch with work in progress by Section X-C, of SAE-ASTM Technical Committee on Automotive Rubber, on stress-relaxation and corrosion of gasketing materials.

Subcommittee VIII on Nomenclature (R. G. Seaman, chairman) has initiated the Tentative Definitions for Elastomers referred to earlier in the report. It will continue to select terms from the Glossary for incorporation in this standard.

Subcommittee IX on Insulating Tape (C. W. Pickells, chairman) has a task group working on the preparation of a moisture-resistance requirement for rubber tapes.

Subcommittee X on Physical Testing (L. V. Cooper, chairman) is concerned with the problem of calibration of testing machines. After consultation with Committee E-1 on Methods of Testing, the chairman appointed two men to serve on Subcommittee I of Committee E-1 to find an answer to this problem.

Subcommittee XI on Chemical Analysis of Rubber Products (W. P. Tyler, chairman) is continuing its work on analytical procedures including solubility methods, copper determination, sulfur determination, improved methods for zinc determination, and a rapid reflux extraction method.

Subcommittee XII on Crude Natural Rubber (N. Bekkedahl, chairman), after recommending the adoption of various ISO TC/45 Methods for Crude Natural Rubber, is now studying rapid methods for the determination of deterioration of natural rubber. It is also concerned with the physical testing of natural rubber and the effect of different mill ratios on the resulting properties. In this study it will cooperate with Subcommittee X on Physical Testing.

Subcommittee XIII on Synthetic Elastomers (B. S. Garvey, Jr., chairman) has several task groups working on the problems of (1) sampling oil and black masterbatches, (2) method for determination of oil content and total extract of SBR and oil masterbatches, (3) method for determination of antioxidant in oil masterbatch. A task group will also work with Subcommittee X on Physical Testing.

Subcommittee XIV on Abrasion Tests for Rubber Products (R. F. Tener, chairman) is considering revision of Methods D 394 including a standard compound for heels and soles.

Subcommittee XV on Life Tests for Rubber Products (G. C. Maassen, chairman) is continuing its work on interlaboratory testing for ozone resistance. Also, it is working with Subcommittee V and Committee D-20 on Plastics on the problems pertaining to aging ovens for highly plasticized rubber compounds and poly(vinyl chloride) compositions.

Subcommittee XVII on Tests of Hardness, Set and Creep (S. R. Doner, chairman) has problems concerning compression set methods and low-temperature use of the durometer.

Subcommittee XIX on Rubber and Rubber-Like Materials in Liquids (F. H. Fritz, chairman) is studying the adequacy of Method D 471 for test temperatures of 300 F. They are working closely with Section IV-H of the SAE-ASTM Technical Committee on the subject of test fluids representing new automotive fuels.

Subcommittee XX on Adhesion Tests (H. H. Irvin, chairman) is continuing its investigations of the effect of environmental conditions on bond strength and on nondestructive and dynamic tests.

Subcommittee XXI on Cements and Related Products (J. F. Anderson, chairman) is investigating methods for nondestructive tests for cement bonds. It is giving consideration to a disk shear test. The problem of a rubber-to-cord adhesion test will be studied.

Subcommittee XXII on Cellular Rubbers (H. G. Bimmerman, chairman) has a problem concerning the variation in results with various indentation machines for measuring the load-deflection characteristics of foamed latex. It is also reviewing the SPI methods for testing urethane and vinyl foams.

Subcommittee XXIII on Hard Rubber (W. J. Dermody, chairman) has task forces working on impact methods and water absorption methods for hard rubber.

Subcommittee XXIV on Coated Fabrics (K. L. Keene, chairman) is continuing its work on abrasion tests and improved methods for testing silicone-coated fabrics for tensile strength.

Subcommittee XXV on Low-Temperature Tests (R. S. Havenhill, chairman) is making progress on the problem of determining the speed of the Scott solenoid impact arm using the ball-thrust method. This will lead to a revision of Method D 746.

Subcommittee XXVI on Processibility Tests (R. H. Taylor, chairman) is continuing its investigations leading to improvements in the two methods using the Mooney viscometer, D 927 – 55 T and D 1077 – 55 T.

Subcommittee XXVII on Resilience (W. A. Frye, chairman) is making a survey to determine the usage of the two tentatives for Yerzley Resilience, Method D 945 – 55 T, and Impact Resilience, Method D 1054 – 55 T. It is also considering the standardization of forced vibration testing machines.

Subcommittee XXVIII on Statistical Quality Control (E. M. Bader, chairman) serves in a consulting capacity to all subcommittees.

Subcommittee XXIX on Compounding Ingredients (A. E. Juve, chairman) continues to work on new standard compounding materials. It is cooperating with Subcommittee XIII in reviewing standard testing formulas for SBR rubbers for incorporation in Methods D 15. Additional work is also being done on standard formulas for testing carbon black.

The SAE-ASTM Technical Committee on Automotive Rubber (C. P. Mullen, chairman) is actively engaged in the preparation of a revised format for the Specifications for Elastomer Compounds for Automotive Applications (D 735 – 58 T). It is cooperating with several other organizations on this problem. In cooperation with Subcommittee XIX it is investigating the use of the higher aromatic gasolines for test fuels.

ISO Technical Committee 45.—The American Group, under the chairmanship of R. D. Stiehler, is actively cooperating with the foreign members and will be directly responsible for participation in the 1959 meeting in New York.

This report has been submitted to letter ballot of the committee, which consists of 257 members; 178 members returned their ballots, of whom 156 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

S. COLLIER,
Chairman.

JOHN J. ALLEN,
Secretary.

REPORT OF COMMITTEE D-12
ON
SOAPS AND OTHER DETERGENTS*

Committee D-12 on Soaps and Other Detergents held one meeting during the year: on March 10 and 11, 1958, in New York City. Three papers were presented at the meeting: "The Analysis of Sodium Triphosphate by Reverse Flow Ion Exchange" by R. H. Kolloff; "The Determination of Alkyl Aryl Sulfonates by Ultraviolet Absorption" by E. W. Blank and W. E. Thompson; and an informal talk by H. W. Zussman which followed up last year's talk on the evaluation of optical brightening agents, bringing the information up to date.

A Committee D-12 Award has been established to honor those persons having performed outstandingly in the field of soaps and detergents. The first recipient was the honorary chairman of Committee D-12, Frederick W. Smither, who is a founding member of Committee D-12. Mr. Smither's pioneering accomplishments and long-continued services to industry and the committee in the development and standardization of test methods were the basis for his award.

The committee consists of 119 members, of whom 57 are classified as producers, 34 as consumers, and 28 as general interest members, with 4 consulting members.

The officers elected for the ensuing term of two years are as follows:

Honorary Chairman, F. W. Smither.
Chairman, J. C. Harris.
Vice-Chairman, W. H. Koch.
Secretary, H. R. Suter.

Advisory Committee G-1, in addition to the officers already listed: M. F. Graham, R. E. Hauber, W. H. Joy, D. Price, A. M. Schwartz, and W. Stericker.

PROPOSED METHODS TO BE PUBLISHED AS INFORMATION

The committee recommends for publication as information only the Method of Test for Sodium Alkylbenzene Sulfonate in Synthetic Detergents By Ultraviolet Absorption, as appended hereto.[1]

NEW TENTATIVES

The committee recommends for publication as tentative the following methods as appended hereto:[2]

Method of Testing:

Detergent Cleaners for Evaluation of Corrosive Effects on Certain Porcelain Enamels.

Methods for:

Sampling and Chemical Analysis of Alkylbenzene Sulfonates,
Testing Detergent Alkylate, and
Sampling and Chemical Analysis of Fatty Alkyl Sulfates.

REVISIONS OF TENTATIVES

The committee recommends revisions as indicated of the following tentatives and their continuation as tentative:

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* Sixty-first Annual Meeting of the Society, June 22–27, 1958.

[1] The Proposed Method appears in the 1958 Compilation of ASTM Standards on Soaps and Other Detergents.

[2] The new and revised tentatives and standards appear in the 1958 Book of ASTM Standards, Part 10, and in the 1958 Compilation of ASTM Standards on Soaps and Other Detergents.

Tentative Definitions of Terms Relating to Soaps and Other Detergents (D 459–57 T):[3] Add the following new definitions:

Artificially Soiled Cloth (sometimes called "Standard Soiled Cloth").—Cloth soiled with one or more materials and used to evaluate the effectiveness of detergents or washing equipment.

Chelating Agent.—A sequestering or complexing agent that, in aqueous solution, renders a metallic ion inactive through the formation of an inner ring structure with the ion.

Complexing Agent.—See Sequestering Agent.

Sequestering Agent.—Any compound that, in aqueous solution, combines with a metallic ion to form a water-soluble combination in which the ion is substantially inactive.

Tentative Methods for Sampling and Analysis of Sodium Triphosphate (D 501–56 T):[4] Revise as appended hereto.[2]

REVISIONS OF STANDARDS, IMMEDIATE ADOPTION

The committee recommends for immediate adoption revisions as indicated of the following standards and accordingly asks for the necessary nine-tenths affirmative vote at the Annual Meeting in order that the revisions may be referred to letter ballot of the Society:

Standard Methods of Sampling and Chemical Analysis of Soaps and Soap Products (D 460–54):[5] Revise as appended hereto.[2]

Standard Methods of Sampling and Chemical Analysis of Alkaline Detergents (D 501–57):[3] Revise as appended hereto.[2]

Standard Methods of Chemical Analysis of Industrial Metal Cleaning Compositions (D 800–45):[5] Revise as appended hereto.[2]

Standard Methods of Chemical Analysis of Soaps Containing Synthetic Detergents (D 820–46):[5] Revise as appended hereto.[2]

REAPPROVAL OF STANDARDS

The committee recommends the reapproval of the following standards which have stood for six or more years without revision:

Standard Specifications for:

Caustic Soda (Anhydrous) (**D 456 – 39**),
Modified Soda (Sesquicarbonate Type) (**D 457 – 39**),
Chip Soap (**D 496 – 51**),
Ordinary Laundry Bar Soap (**D 497 – 52**),
Powdered Soap (Nonalkaline Soap Powder) (**D 498 – 51**),
White Floating Toilet Soap (**D 499 – 48**),
Built Soap, Powdered (**D 533 – 44**),
Alkaline Soap Powder (**D 534 – 42**),
Palm Oil Solid Soap (**D 535 – 52**),
Palm Oil Chip Soap (**D 536 – 52**),
Olive Oil Solid Soap (**D 592 – 42**),
Salt-Water Soap (**D 593 – 42**),
Sodium Sesquisilicate (**D 594 – 41**),
Tetrasodium Pyrophosphate (Anhydrous) (**D 595 – 45**),
Olive Oil Chip Soap (**D 630 – 42**),
Compound Chip Soap (with Rosin) (**D 690 – 48**),
Compound Powdered Soap (Granulated, with Rosin) (**D 691 – 44**),
Liquid Toilet Soap (**D 799 – 51**),
Sodium Bicarbonate (**D 928 – 52**), and
Borax (**D 929 – 50**).

Standard Method of:

Total Immersion Corrosion Test of Water-Soluble Cleaners (**D 930 – 49**), and
Test for Particle Size of Soaps and Other Detergents (**D 502 – 39**).

EDITORIAL CHANGES

The committee recommends editorial changes as indicated in the following standards:

Standard Specifications for Olive Oil Solid Soap (D 592–42):[5]

Table I.—Add a reference to Footnote *b* under type B in the line "Titer of mixed fatty acids prepared from the soap."

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[3] 1957 Supplement to Book of ASTM Standards, Part 7.

[4] 1956 Supplement to Book of ASTM Standards, Part 7.

[5] 1955 Book of ASTM Standards, Part 7.

Standard Specifications for Olive Oil Chip Soap (D 630–42):[5]

Table I.—Add a reference to footnote *b* as indicated above for Specifications D 592.

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[6]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee T-2 on Analysis of Soaps and Synthetic Detergents (J. C. Harris, chairman):

Task Group 3 on Bibliographical Abstracts of Analytical Methods for Surfactants (J. C. Harris, chairman). Copy for *Special Technical Publication No. 150-B* (1933–1957) has been completed.

Task Group 8 on Determination of Carboxymethyl Cellulose (J. L. Darragh, chairman).—A procedure has been subjected to cooperative analysis. Lack of agreement between laboratories indicated the need for improvement, and this is contemplated for the coming year.

Task Group 10 on Sulfate Content in Presence of Phosphate (H. C. Bennett, chairman).—In addition to the action on Methods D 460–54, the task group will continue its investigation of a yet more improved procedure.

Task Group 11 on Determination of Active Ingredient by Ultraviolet Absorption (E. W. Blank, chairman).—In addition to the development of the proposed method referred to earlier in the report, the data developed in support of the procedure will be drawn up for publication purposes to give it wider recognition.

Task Group 14 on Rapid Determination of Moisture by Infrared Lamp (O. L. Sherburne, chairman).— Cooperative work has resulted in a test method to be subjected to cooperative testing.

Task Group 15 on Determination of Active Ingredient in Soaps Containing Synthetic Detergents by Cationic Titration (L. E. Weeks, chairman).—Cooperative work has resulted in agreement on a procedure, but satisfactory characterization of an anionic standard detergent sample is needed. Plans are made to isolate the standard in a highly purified condition for determination of combining weight.

Task Group 20 on Total Active Ingredient (C. A. Cohen, chairman).—Cooperative analyses by ten laboratories testing a new procedure gave good reproducibility except for combining weight. This procedure will be corrected and new samples evaluated.

Task Group 21 on Detergent Alkylate (J. C. Harris, chairman).—In addition to the action on the characterization of detergent alkylate, subgroups submit the following reports:

Sub-Group B on Molecular Weight (C. A. Cohen, chairman).—A set of standard samples is now available for determination of molecular weight by a refractivity intercept method. Cooperative tests of the procedure will be carried on.

Sub-Group C on Acid Wash Color (R. C. Stillman, chairman).—Using Method D 848–47, it was found that results varied widely. Test conditions will be made more precise and cooperative effort continued.

Sub-Group D on Standard Sulfonation Procedure and Analysis of Product (C. A. Cohen, chairman).—A new flask design has been completed, and cooperative effort is anticipated by this fall.

Subcommittee T-4 on Analysis of Inorganic Alkaline Detergents (M. V. Trexler, temporary chairman).—The task group headed by M. V. Trexler and charged with the comparison of the ion exchange method for identification and analysis of sodium triphosphate analyzed statistically the data from this work with that on the "cobalt" and "paper chromatographic" methods. These data showed the latter method to be the most precise and accurate of the three.

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[6] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

A new task group with C. H. Russell as chairman was established to evaluate a newly described reverse-flow ion-exchange technique.

Subcommittee T-5 on Physical Testing (M. G. Kramer, chairman).—The activities of the several task groups follow:

Task Group 1 on Measurement of Soil Redeposition (A. R. Martin, chairman).—Data from the cooperative testing program have been compiled and analyzed statistically. A report is in process of review.

Task Group 2 on Measurement of Wetting Power (C. A. Gerardi, chairman).—Another survey is to be undertaken to determine which of existing methods should be studied.

Task Group 3 on Measurement of Reflectance of Test Fabrics (R. L. Liss, chairman).—Results of the earlier tests are being compiled and will be studied for guidance to future action.

Task Group 5 on Method for Evaluating Brightening Agents (M. S. Furry, chairman).—Reorganization of the group will be made in an attempt to develop methods for application and evaluation.

Task Group 8 on Methods for Evaluation of Rug Cleaning Detergents (J. W. Rice, chairman).—A recently built soiling machine will be used to furnish rug samples for use in the evaluation method.

Task Group 9 on Methods for Determining the Cloud Point of Surfactants (R. L. Mayhew, chairman).—Existing methods will be collected for use in designing a procedure for cooperative laboratory evaluation.

Task Group 10 on Methods for Evaluating Detergents in Dishwashing (E. V. Plock, chairman).—A survey of the membership of Subcommittee T-5 resulted in the information that five areas of testing should be considered: Detergency, corrosion of metals, chemical attack and staining of dishes, foaming, and shelf life. Test methods will be accumulated.

Task Group 11 on Studies of Primary Soil Deposition (A. M. Schwartz, chairman).—An annotated bibliography has been started which should be completed within the coming year.

Subcommittee T-6 on Analysis of Metal Cleaners (H. A. Kafarski, chairman):

Task Group A (R. O. Kageff, chairman).—The scope of action of the group was revised to include methods and typical materials for cleaning aluminum trim.

Task Group B on Metal Cleaners—Industrial Power Spray Washers (H. A. Kafarski, chairman).—Shop and laboratory methods are to be investigated for effectiveness and application of cleaning compositions.

Subcommittee S-2 on Specifications for Soaps and Synthetic Detergents (W. H. Joy, chairman).—A task group with R. L. Liss as chairman has been established to develop specifications for synthetic detergents along the lines of Federal Specifications now existent.

Subcommittee S-4 on Specifications for Inorganic Alkaline Detergents (W. Stericker, chairman).—An additional grade of soda ash is to be added to Specifications D 458 during the coming year. Preliminary limits for sodium triphosphate were discussed and are to be drawn up in specification form.

This report has been submitted to letter ballot of the committee, which consists of 119 members; 48 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

J. C. HARRIS,
Chairman.

H. R. SUTER,
Secretary.

REPORT OF COMMITTEE D-13
ON
TEXTILE MATERIALS*

Committee D-13 on Textile Materials held two general meetings during the year: in New York City, N. Y., on October 15 to 18, 1957, and in Washington, D. C., on March 18 to 21, 1958. Numerous subcommittees met in conjunction with the main committee, and the practice of devoting an extra day to task group meetings was continued.

During the general session at the October meeting, three papers dealing with the subject of textile fiber test methods and standards developed by Committee D-13 were presented by Dorothy Patt, American Cyanamid Co.; William Poisson, Allied Chemical and Dye Corp.; and George Wham, Good Housekeeping Inst.

At the March meeting, the Harold DeWitt Smith Memorial Medal for 1958 was presented to Stephen J. Kennedy, Research Director, Textile, Clothing and Footwear Division, Headquarters, Quartermaster Research and Development Center, U. S. Army, Natick, Mass. The citation on the Scroll presented to Mr. Kennedy reads as follows:

> To
>
> Stephen Jay Kennedy
>
> In recognition of his long and outstanding service to the Army and to the country through the improvement of textile materials; his resourcefulness in initiating and guiding a broad program of research that led to the development of textiles to meet world-wide environmental requirements for military uses; and his success in enlisting the cooperation of scientists and industry in the design and development of clothing, footwear, and shelter, which increased the comfort and protection of the American soldier and which resulted in materially improved military efficiency, increased savings to the government, and lasting benefit to the civilian economy.

During the year a survey of test methods and standards sponsored by Committee D-13 was made, as a result of which consideration is being given to substituting reference citations for some of the standards that are not frequently used.

The committee approved enlarging the scope of Subcommittee B-6 which henceforth will be known as the "Coordinating Committee for ISO and Foreign Standards." During the year the chairman of this committee, A. G. Scroggie, has continued to work with ASA Committee L-23 in conjunction with international standardization of textile test methods.

The following appointments have been made during the year: Burt Johnson, chairman of Subcommittee A-1 on Cotton Fibers, to replace G. S. Buck, Jr. (resigned); Lyman Fourt, formerly chairman of Subcommittee B-9 on Fabric Test Methods, General, to be chairman of Subcommittee B-1 on Chemical and Performance Test Methods to replace M. S. Staples (resigned); N. J. Abbott, chairman of Subcommittee B-9 on Fabric Test Methods, General, to replace Lyman Fourt; D. M. Scott, chairman of Subcommittee C-3 on Membership, to replace G. E. Hopkins (resigned); E. N. Ditton, chairman of Subcommittee B-3 on Atmospheric Conditions and Regain, to replace D. H. Spitzli (resigned); J. T. Taylor, chairman of Subcommittee A-10 on Non-Woven Fabrics, Section II, to replace J. K. Sumner; and T. L. Rusk, Secretary of Subcommittee B-2 on Nomenclature and Definitions, to replace Robert Graham.

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* Presented at the Sixty-first Annual Meeting of the Society, June 22–27, 1958.

The officers elected for the ensuing term of two years are as follows:

Chairman, B. L. Whittier.

First Vice-Chairman, A. G. Scroggie.

Second Vice-Chairman, Ruby K. Worner.

Secretary, H. A. Ehrman.

RECOMMENDATIONS AFFECTING STANDARDS

The committee is presenting for publication 1 proposed method as information, 8 new tentatives, the revision of 5 tentatives, the immediate adoption as standard of revisions of 14 standards, the revision and reversion to tentative of 2 standards, the adoption of 10 tentatives as standard, the withdrawal of 9 standards, and the tentative revision of one standard.

The standards and tentatives affected, together with the revisions recommended, are listed in detail in the Appendix.[1]

All other standards and tentatives not specifically referred to are being studied by the respective subcommittees and it is recommended that they be continued in their present status.

The recommendations appearing in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[2]

This report has been submitted to letter ballot of the committee, which consists of 378 voting members; 145 members returned their ballots, of whom 135 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

B. L. WHITTIER,
Chairman.

H. A. EHRMAN,
Secretary.

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[1] See p. 371.

[2] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

APPENDIX

RECOMMENDATIONS AFFECTING STANDARDS FOR TEXTILE MATERIALS

In this Appendix are given proposed revisions in certain standards and tentatives covering textile materials. These standards and tentatives appear in their present form in the 1955 Book of ASTM Standards, Part 7, or in the 1956 and 1957 Supplements, Part 7.

PROPOSED METHOD TO BE PUBLISHED AS INFORMATION

The committee recommends for publication as information only the Proposed Method of Test for Dimensional Change of Knitted Fabrics in Laundering and Dimensional Restorability of the Laundered Fabric, appended hereto.[1]

NEW TENTATIVES

The committee recommends for publication as tentative the following methods as appended hereto:[2]

Methods of Test for:

Industrial Asbestos Tapes
Heat Aging of Asbestos Textiles
Woven Asbestos Cloth
Linear Density of Textile Fibers by the Vibroscope
Moisture in Wool by Oven Drying
Extractable Matter in Oven Dried Wool
Fiber Length of Wool
Breaking Strength of Yarn by Skein Method

REVISION OF TENTATIVES

The committee recommends revisions as indicated of the following tentatives and their continuation as tentative:

Tentative Method and Tolerances for Yarn Containing Wool (D 1285 – 56 T):

Section 10(b).—Change the tolerances for yarn number of worsted yarn to read as follows:

	Yarn Number Tolerances
Single yarn, coarser than No. 23	±4.5
Single yarn, No. 23 and finer	±4.0
Plied yarn, all numbers	±5.5

The new tolerances have been obtained by using a 95 per cent level of probability quoted elsewhere in the standard, in lieu of a 99.7 per cent level of probability.

Tentative Methods of Test for Twist in Single Spun Yarns (Untwist-Twist Method) (D 1422 – 56 T):

Section 6.—Change to read as follows:

6. (*a*) *Number of Tests*.—Unless otherwise agreed upon, the number of test specimens shall be such as to give a precision of the mean of the test results of ±5 per cent at a probability level of 95 per cent:

$$n = 0.154 V^2$$

where:

n = number of test specimens, and
V = coefficient of variation of individual test results determined from extensive past records on similar yarns.

(*b*) If V is not known, 20 tests (2 from each of 10 bobbins) shall be performed when the specimen length is 10 in., and 30 tests (3 from each of 10 bobbins) when the specimen length is 5 in. These numbers of tests have been calculated to give the required precision of ±5 per cent for $V = 11.5$ per cent (10 in. specimens), or $V = 14.0$ per cent (5-in. specimens).

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[1] The proposed method of test appears in the 1958 Compilation of ASTM Standards on Textile Materials.

[2] The new and revised tentatives and standards appear in the 1958 Book of ASTM Standards, Part 10.

NOTE 3.—The values assumed for V in this section are somewhat higher than are likely to be encountered in practice. Therefore the formula in Paragraph (*a*) will in most cases give a lower number of tests than are specified in Paragraph (*b*). To use the formula in Paragraph (*a*), the V applying to the yarn under test must be known.

Tentative Recommended Practice for Designation of Yarn Construction (D 1244 – 57 T):

Revise as appended hereto.[2]

Tentative Method of Test for Twist in Single Spun Yarns (Direct-Counting Method) (D 1423 – 56 T):

Revise as appended hereto.[2]

Methods of Testing and Tolerances for Rayon Tire Cord (D 885 – 56 T):

Replace these methods by the Proposed Methods of Testing and Tolerances for Tire Cords from Man-Made Fibers published as information since November, 1956,[3] revised as follows:

Section 9.—Change to read as follows:

9. *Elongation*.—The average percentage of elongation of the cord shall not vary by more than the amount shown in the following table of the specified elongation:

	At 10 lb	At Break
Rayon.....	±20 per cent	±15 per cent
Nylon and Polyester.	±10 per cent	±10 per cent

Section 17(*b*).—Change to read as follows:

(*b*) *Calculation*.—The linear density shall be expressed in denier or tex units as calculated from one of the following formulas:

Denier at a commercial moisture regain = $\dfrac{9000\,D \times K}{L}$

Tex at a commercial moisture regain = $\dfrac{1000\,D \times K}{L}$

Denier at actual moisture regain = $\dfrac{9000\,C}{L}$

Tex at actual moisture regain = $\dfrac{1000\,C}{L}$

where:

D = oven-dry weight of specimen in grams,
C = conditioned weight of specimen in grams,
L = length of specimen in meters (or yards × 1.09), and
K = factor for commercial regain.

Section 18.—Change the last sentence of Paragraph (*a*) to read as follows: "A tension device or weight shall be provided which will apply an actual pretension of 3 to 6 oz (85 to 170 g) to the specimen in the test area.

Change Paragraph (*b*) to read as follows:

(*b*) *Procedure*.—Remove the specimen from the sample and handle in such a manner that no change in twist can occur prior to closing the jaws of the clamps on the testing machine. Place the specimen between the jaws of the two clamps, close one of the clamps, and apply a pre-tension load to the unclamped end which will result in the specified tension in the test area. Then close the clamp and break the specimen. Read the breaking strength in pounds or kilograms from the chart. Discard specimens which break in the jaws or within $\frac{1}{8}$ in. of the nips of the jaws.

NOTE 1.—The actual pre-tension in the test area is commonly different than that amount applied externally by a tension device or weight due to friction losses in the clamp.

NOTE 2.—The actual pre-tension can be measured by the use of strain gages. Other tension measuring instruments with sufficient accuracy can be used, provided the specimen is threaded through the instrument prior to being placed in the second clamp. This is necessary as many instruments require appreciable displacement of the specimen. The actual tension shall be measured with the jaws closed.

NOTE 3.—The specified tension shall be used in all cases involving arbitration of test data. If a testing laboratory is not equipped to measure the actual tension an approximation can be obtained by attaching a 6-oz load to the unclamped cord prior to closing the second clamp.

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[3] See compilation of ASTM Standards on Textile Materials, Appendix X, p. 737 (November, 1957).

REVISION OF STANDARD AND REVERSION TO TENTATIVE

The committee recommends revisions as indicated of the following standards and their reversion to tentative:

Standard Specification and Methods of Test for Asbestos Tape for Electrical Purposes (D 315 – 52):

Revise as appended hereto.[2]

Recommended Practice for Designation of Linear Density of Fibers, Yarns, and Other Textile Materials in Universal Units (D 861 – 52):

Revise as appended hereto.[2]

ADOPTION OF TENTATIVES AS STANDARD WITHOUT REVISION

The committee recommends that the following tentatives be approved for reference to letter ballot of the Society for adoption as standard without revision:

Tentative Methods of Test for:

Fineness of Wool (**D 419 – 55 T**),
Fiber Length of Wool Top (**D 519 – 55 T**),
Tensile Strength of Wool Fiber Bundles (**D 1294 – 56 T**),
Wool Content of Raw Wool (Commercial Scale) (**D 1334 – 54 T**). This is subject to the revision recommended in Method D 1060.
Determination of Build-up and Spread of Glass Yarn as Wrap (Serving) or Braid over Electrical Conductors) (**D 1332 – 54 T**), and
Determining Specific Area of Immature Ratio of Cotton Fibers (Arealometer Method) (**D 1449 – 55 T**).

Recommended Practice for:

Interlaboratory Testing of Textile Materials (**D 990 – 54 T**),
Tolerances for Filament Acetate Yarns (**D 1235 – 52 T**),
Tolerances for Filament Nylon Yarns (**D 1236 – 52 T**), and
Tolerances for Filament Rayon Yarns (**D 1237 – 52 T**).

REVISION OF STANDARDS, IMMEDIATE ADOPTION

The committee recommends for immediate adoption revisions as indicated of the following standards, and accordingly asks for the necessary nine-tenths affirmative vote at the Annual Meeting in order that the revisions may be referred to letter ballot of the Society:

Method of Core Sampling of Raw Wool in Packages for Determination of Percentage of Clean Wool Fiber Present (D 1060 – 56):

Section 1.—Renumber the present Section 1 as "1 (*a*)" and add a new Paragraph (*b*) to read as follows:

> (b) This method also provides a procedure for calculating the size of sample required for a desired precision when certain statistical data pertaining to the lot under consideration are available.

Section 2(a).—Add to the end of the first sentence: "subject to the following restrictions." Delete the second sentence. Change "(Note 4, Section 6)" to "(Note 4, Appendix)."

Table I.—Remove this table, including the footnotes, from the body of the method and place in the Appendix as indicated.

Section 4(c).—Add at the end of the third sentence: "and normal to that surface."

At the beginning of the fourth sentence, insert: "Packages shall be so positioned that." In the same sentence, change "shall be only" to "will be only."

Section 5(e).—Change to read as follows:

> (*e*) *Estimates of the Variances.*—The true values of σ_w^2 and σ_b^2 for any given lot of wool will not in general be known. Reliable estimates of their approximate values based on previous experience with similar lots of wool, or obtained by a preliminary test or otherwise, shall be used. The Appendix to this method lists the observed average values of σ_w and σ_b for lots of several types of wool as they appeared in the commerce of the United States prior to the adoption of this revision of the method. When no reliable estimates are available, it is recommended that the conservative value of 5.0 be used for both σ_w and σ_b in Eq. 1 when sampling is performed in accordance with this method.

Section 6.—Delete this section, including Notes 4 and 5.

Section 7.—Change to read as follows:

7. Table I gives the values of n calculated from Eq. 1 for selected pairs of values of σ_w and σ_b and for selected lot sizes, N, and numbers of cores per package, k, for a fixed sampling precision of ±1.0 at a probability level of 0.95.

Table II.—Renumber as Table I. Change the heading to read: "Values of n Calculated from Eq. 1^a for a Precision of ±0.1 at a Statistical Probability of 0.95, for Selected Value of σ_w, σ_b, and k."

New Appendix.—Add to the method the following Appendix:

APPENDIX

CLASSIFICATION OF WOOLS FOR SAMPLING PURPOSES

Committee studies have shown that the values of σ_w and σ_b listed in Table II are good estimates of the average values for lots of substantially undamaged packages (Note 4) of the respective classes of wool as they appeared in the commerce of the United States prior to the adoption of this revision of the method, when the lots conformed with the restrictions in Section 2(*a*) and were sampled in accordance with the procedures described in the method. Any subsequent material changes that may have occurred in the practices of preparing or packaging any of the wools or grouping packages into lots, may have resulted in changes in the corresponding values of σ_w and σ_b, so that the tabulated values may no longer be suitable for use in calculating a sampling schedule for the affected wools.

When the true values of σ_w and σ_b for a given lot of wool are any pair of the values shown in Table II, the sampling schedules may be conveniently obtained directly from Table I in the method.

NOTE 4.—The data on Table II do not apply to lots containing packages that have been materially damaged by water, oil, or other agent unless these packages are first removed from the lot for separate evaluation. Normally soiled or torn coverings of themselves do not indicate material damage for sampling purposes.

TABLE II.—Include all of Table I of the method, with the following changes:

1. Omit footnote *b* and the reference thereto.
2. Indent the words "white" "colored" under "India."
3. Change the value of σ_b opposite Pakistan from 2.0 to 5.0.

Methods of Testing Nonwoven Fabrics (D 1117 – 57):

New Sections.—Add the following methods for flexural rigidity and dimensional change as new Sections 12 and 13:

12. Flexural Rigidity.—The stiffness of nonwoven fabrics is determined by the methods used for woven fabrics under D 1388 – 55 T Method A, Cantilever Test. The words "machine direction" and "cross machine direction" should be substituted for "warp" and "filling" respectively.

13. Dimensional Change: (*a*) *Purpose and Scope.*—This method is intended to be used for determining the changes in dimensions, shrinkage or stretching, of nonwoven fabrics which take place when the fabric is subjected to any treatment such as laundering or dry cleaning, etc. which could cause such changes.

(*b*) *Test Specimens.*—The specimen before and after treatment shall be conditioned for four hours at 65% (±2%) relative humidity and 70 F (±2 F). The specimen is laid out without tension on a flat surface, care being taken that the fabric is smooth and free from wrinkles and creases. Three distances, each 18 inches long, in both the machine and across the machine directions, shall be marked off. These distances shall be parallel to these directions and approximately 6 inches apart and all of them at least one inch from the edges of the sample. Satisfactory marks may be made with indelible ink (or any other marking material resistant to the treatment to which the fabric will be subjected) using a fine pointed or ball point pen, or fine threads may be sewn into the fabric, or any other suitable marking device may be used. These markings should be made on the fabric immediately following the conditioning of the specimen and before treating it.

(*c*) *Drying.*—If the treatment prior to measuring shrinkage involved wetting the fabric with water (as in laundering), or with a solvent (as in dry cleaning), the specimen must first be dried. If wet with water, squeeze out the excess gently by hand so as not to distort the fabric. Wringing by hand or with squeeze rolls is not permissible, as either may distort the fabric. Spread out the specimens on a horizontal screen or similar perforated surface and allow to dry at room temperature. The drying process may be speeded up by directing a current of air, either slightly heated or at room temperature, on the fabric. The specimen may also be hydro-extracted and dried in an oven if the temperature of the oven is held at a level known to be harmless to any of the ingredients in the fabric. The dried specimens are then dampened with water and allowed to stand 5 min.

(*d*) *Pressing.*—Press the specimens either with a flat-bed press or a hand iron, taking care in handling them to avoid any strain on the fabric. Smooth each specimen to remove wrinkles as well as possible before pressing. When a hand iron is used, do not slide the iron back and forth on the specimen, but simply press down upon it in a manner simulating the action of a flat-bed press. The temperature used must be suitable for the fiber, binder and other materials in the fabric. Allow the specimen to cool to room temperature before measuring it.

(*e*) *Evaluation.*—The pressed specimen is laid out without tension on a flat surface, care being taken that the fabric is smooth and free from wrinkles or creases. The distances which were marked off are measured again. The shrinkages in the machine direction and across the machine direction are calculated separately as the averages obtained from the marked distances. The shrinkage is the change in the dimensions measured and is expressed either as a percentage of the dimensions before treatment or in inches per yard.

Definitions of Terms Relating to Textile Materials (D 123 – 55):

Change the definition of the term "Tape, Woven" to read as follows:

Tape, Woven, n.—A woven narrow fabric, made principally of cotton, linen, and/or man-made fibers, but not containing rubber or other similar elastomer, and weighing less than 15 oz per sq yd (2.625 lb per 100 yd per in. of width).

Standard Specifications and Methods of Test for Asbestos Roving for Electrical Purposes (D 375 – 52):

Section 5.—Change this section to read as follows:

5. *Magnetic Iron Rating.*—Roving shall be made in two varieties, ferrous and non-ferrous, the magnetic iron ratings of which shall conform to the following requirements:

	Magnetic Iron Rating, max
Ferrous roving	3
Non-ferrous roving:	
Underwriters' Grade	0.75
Grade A	1

Sections 10, 11, 12, and 13.—Delete these sections.

Section 14.—Change to read as follows, renumbering to Section 10:

10. *Magnetic Iron Rating.*—The magnetic iron rating shall be determined in accordance with the Method of Test for Magnetic Rating of Asbestos Used for Electrical Purposes (ASTM Designation: D 1118).

Standard Specifications and Methods of Test for Asbestos Lap (D 1061 – 57):

Section 4.—Change this section to read as follows:

4. *Magnetic Iron Rating.*—Asbestos lap shall be made in two varieties, ferrous and non-ferrous, the magnetic iron rating of which shall conform to the following requirements:

	Magnetic Iron Rating, max
Ferrous lap	3
Non-ferrous lap:	
Underwriters' Grade	0.75
Grades A, AA, and AAA	1

Section 10.—Delete this section.

Renumber Sections 10 and 11, accordingly.

Standard Methods of Testing and Tolerances for Glass Yarns (D 578 – 52):

Section 5(b).—Change the table to read as follows:

Turns per Inch	Permissible Variations, plus or minus, per cent
0.5–1.0	30
1.1–2.5	20
2.6–5.0	15
5.1–10.0	10
10.1 up	5

Standard Methods of Testing and Tolerances for Single Jute Yarn (D 541 – 52):

Section 12(b).--Add the following paragraph:

By mutual agreement, testing machines operated at constant rate of load (inclined plane), or using strain gages, may be used if their precision and accuracy are at least equal to the pendulum type applicable to the same conditions. Results from the three types of machines are not interchangeable, unless the time to break (not rate of extension, nor rate of load) are identical within about ±2 sec.

Standard Methods of Testing and Tolerances for Jute Rove and Plied Yarn for Electrical and Packing Purposes (D 681 – 52):

Section 11(b).—Add the same paragraph as recommended above for Method D 541.

Standard Methods of Testing and Tolerances for Rope Made from Bast and Leaf Fibers (D 738 – 52):

Section 9(c).—Add the same paragraph as recommended above for Method D 541.

Standard Methods of Testing and Tolerances for Spun, Twisted, or Braided Products Made from Flax, Hemp, Ramie, or Mixtures Thereof (D 739 – 52):

Section 16(b).—Add the same paragraph as recommended above for Method D 541.

Standard Methods of Testing Single Kraft Yarn (D 1057 – 51):

Section 6(b).—Add the same paragraph as recommended above for Method D 541.

Standard Methods of Testing Twine Made from Bast and Leaf Fibers (D 1233 – 52):

Section 12(b).—Add the same paragraph as recommended above for Method D 541.

Standard Method of Test for Fineness of Cotton Fibers by Micronaire (D 1448 – 56):

Section 8.—Change "Micronaire units" to "Micronaire reading."

Standard Methods of Test for Water Resistance of Textile Fabrics (D 583 – 54):

New Sections 13 to 15.—Add the following dynamic absorption test as Method II as an alternate test to the present method in Sections 10 to 12. Designate the present method as Method I. Renumber the remaining sections.

DYNAMIC ABSORPTION TEST, METHOD II

(AATCC METHOD 70A—1958)

13. *Apparatus and Materials:* (*a*) *Launder-Ometer*, or similar machine, operating at 42 rpm with provision for mounting metal specimen containers (See Paragraph (*b*)) horizontally at right angles to the shaft. Provision shall be made for maintaining the initial temperature of the jars throughout the test.[a]

(*b*) *Standard stainless-steel specimen containers*, $3\frac{1}{2}''$ in diameter by 8″ long.[8]

(*c*) *Stainless steel balls*, $\frac{1}{4}''$ in diameter.[8]

(*d*) *Wringer*, motor-driven, household laundry type, conforming to requirements in Paragraph 10 (*b*).

(*e*) *Laboratory balance*, accurate to 0.1 g.

(*f*) *Blotting paper*, cut to 4″ x 4″, and conforming to requirements in Paragraph 10 (*c*).

14. *Test Specimens*.—Prepare two sets of ten square pieces, each 3″ x 3″, cut on a 45° bias from the conditioned fabric and with the loose corner yarns removed. Ten pieces shall constitute one specimen.

15. *Procedure:* (*a*) Place 300 ml. of water (see Section 3) and 200 steel balls into each of two standard stainless steel containers. Roll together a specimen consisting of ten conditioned pieces and weigh to the nearest 0.1 g. Repeat with the second specimen of ten pieces from the same sample. Place each specimen, one piece at a time, into a separate stainless steel container. (Crumple each piece before placing it in the container.)

Mount the containers in the Launder-Ometer and run for 20 minutes at 80 ± 2 F. (27 ± 1 C.). At the end of 20 min, quickly remove the pieces of one specimen, one at a time, pass each piece through the wringer once without blotters, with one edge of the piece parallel to the rolls of the wringer; then immediately pass it through the wringer between two unused blotters. Leave the piece between the blotters until the ten pieces from one set have been through the wringer; then remove the ten pieces from the blotters, roll them together again and weigh as before, protecting the pieces from air drying as much as possible during these operations.

(*b*) Repeat the squeezing, blotting, and weighing procedure described in Paragraph (*a*) for the second specimen of ten pieces.

(*c*) Calculate the dynamic absorption for each specimen as follows:

$$\text{Dynamic absorption, per cent} = \frac{\text{Final wt. of specimen} - \text{original wt. of specimen}}{\text{Original wt. of specimen}} \times 100$$

(*d*) The dynamic absorption of the fabric shall be the average of the results obtained from the specimens tested and shall be reported to the nearest 1.0 per cent.

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[a] Equipment and material meeting these specifications are manufactured by Atlas Electric Devices Co., 4114 N. Ravenswood Ave, Chicago 13, Ill.

A description of the Launder-Ometer and accessories is given in the AATCC Technical Manual and Year Book.

TENTATIVE REVISION OF STANDARD

The committee recommends as a tentative revision of the Standard Definitions of Terms Relating to Textile Materials (D 123 – 55) the revised and new tentative definitions appended hereto,[2] including the revised list on fabric defect terminology.

WITHDRAWAL OF STANDARDS

The committee recommends the withdrawal of the following standards since they have been combined and are replaced by the proposed Tentative Specification and Methods of Test for Woven Asbestos Cloth:

Standard Method of Testing Woven Asbestos Cloth (**D 577 – 52**)
Standard Specifications for Woven Asbestos Cloth (**D 677 – 50**)

The committee recommends the withdrawal of the following ASTM methods. These methods were originally developed by the Technical Committee on Research of the American Association of Textile Chemists and Colorists. With the withdrawal of the ASTM standards it is also recommended that appropriate references to the AATCC methods be included in the Book of ASTM Standards and in the annual compilations of ASTM Standards on Textile Materials:

Standard Methods of Test for:

Colorfastness to Commercial Laundering and to Domestic Washing of Cotton and Linen Textiles (**D 435 – 42**),
Colorfastness of Dyed or Printed Wool, Silk, Rayon, or Acetate Fabrics to Laundering or Domestic Washing (**D 436 – 37**),
Colorfastness to Light of Textiles (**D 506 – 55**),
Resistance of Textile Fabrics and Yarns to Insect Pests (**D 582 – 54**),
Evaluating Compounds Designed to Increase Resistance of Fabrics and Yarns to Insect Pests (**D 627 – 54**),
Colorfastness of Dyed Acetate to Atmospheric Fumes (**D 682 – 52**), and
Resistance of Textile Materials to Microorganisms (**D 684 – 57**).

STANDARDS AND TENTATIVES CONTINUED WITHOUT REVISION

The committee recommends that the standards under its jurisdiction which have not been revised within the past six years, and also the tentatives that have not been revised within the past two years be continued in their present status without change.

PUBLICATION ON EXTENDED INTERVAL BASIS

Occasionally there are methods that are not too widely used and the committee believes that in such instances it is not necessary to republish these annually in the Compilation of ASTM Standards on Textile Materials. Subcommittee A-1 on Cotton Fibers has recommended that the following methods under its jurisdiction be published only on an extended interval basis but that in those editions of the compilation which do not include the method, a reference be added to the earlier edition in which it appeared:

Method of Test for Maturity of Cotton Fibers (Polarized-Light Method) (**D 1450 – 57**), and

Test for Strength of Cotton Fibers by Chandler Bundle Method (**D 414 – 40 T**) which was discontinued in 1955.

REPORT OF COMMITTEE D-14
ON
ADHESIVES*

Committee D-14 on Adhesives held two meetings during the year: at the Sheraton Hotel, Philadelphia, Pa., on October 31 and November 1, 1957, and at the U. S. Forest Products Laboratory, Madison, Wis., on April 2, 3, and 4, 1958. On the third day of the April meeting a group of members toured the Forest Products Laboratory.

The committee consists of 127 members, of whom 42 are classified as producers, 63 as consumers, and 22 as general interest or consulting members.

Three new subcommittees have been established—Subcommittee X on Wood Adhesives, Subcommittee XI on Metal Bonding Adhesives, and Subcommittee XII on Adhesives for Plastics.

The officers elected for the ensuing term of two years are as follows:

Chairman, J. E. Rutzler, Jr.
Vice-Chairman, A. A. Marra.
Secretary, J. J. Lamb.
Membership Secretary, W. W. Sederlund.

NEW TENTATIVES

The committee recommends for publication as tentative the following methods and specifications as appended hereto:[1]

Methods of Test for:

Bonding Permanency of Water or Solvent Soluble Liquid Adhesives for Labeling Glass Bottles,
Nonvolatile Content of Phenol, Resorcinol, and Melamine Adhesives,
Measuring the Hydrogen Ion Concentration of Dry Adhesive Films,
Water Absorptiveness of Paper Labels, and
Filler Content of Phenol, Resorcinol, and Melamine Adhesives.

Specification for:

Adhesives for Automatic Machine Labeling of Glass Bottles.

REVISIONS OF TENTATIVES

The committee recommends revisions as follows of the Tentative Method of Test for Effect of Moisture and Temperature on Adhesive Bonds (**D 1151 - 56 T**):[2]

Table I.—Change to read as indicated in the accompanying Table I.

TENTATIVE REVISIONS OF STANDARD

The committee recommends tentative revisions of the Standard Definition of Terms Relating to Adhesives (**D 907 - 55**),[3] consisting of the following new definitions:

Amylaceous, adj.—Pertaining to, or of the nature of, starch; starchy.

Consistency, n.—That property of a liquid adhesive by virtue of which it tends to resist deformation.

NOTE.—Consistency is not a fundamental property but is comprised of viscosity, plasticity, and other phenomena. (See also Viscosity, Viscosity Coefficient, and Consistency Coefficient.)

Flow, n.—Movement of an adhesive during the bonding process, before the adhesive is set.

Modifier, n.—Any chemically inert ingredient added to an adhesive formulation that changes its properties. (See Filler, Plasticizer, and Extender.)

Penetration, n.—The entering of an adhesive into an adherend.

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* Presented at the Sixty-first Annual Meeting of the Society, June 22–27, 1958.

[1] The new tentatives appear in the 1958 Book of ASTM Standards, Part 6.

[2] 1956 Supplement to Book of ASTM Standards, Part 7.

[3] 1955 Book of ASTM Standards, Part 7.

TABLE I.—STANDARD TEST EXPOSURE.
(New Table I for Method D 1151)

Test Exposure Number	Temperature[a]		Moisture Conditions
	deg Cent	deg Fahr	
1	−57	−70	As conditioned
2	−34	−30	As conditioned
3	−34	−30	Presoaked[b]
4	0	32	As conditioned
5	23	73.4	50 per cent relative humidity
6	23	73.4	Immersed in water
7	38	100	88 per cent relative humidity
8	63	145	Oven, uncontrolled humidity
9	63	145	Over water[c]
10	63	145	Immersed in water
11	70	158	Oven, uncontrolled humidity
12	70	158	Over water[c]
13	82	180	Oven, uncontrolled humidity
14	82	180	Over water[c]
15	100	212	Oven, uncontrolled humidity
16	100	212	Immersed in water
17	105	221	Oven, uncontrolled humidity
18	149	300	Oven, uncontrolled humidity
19	204	400	Oven, uncontrolled humidity
20	260	500	Oven, uncontrolled humidity
21	316	600	Oven, uncontrolled humidity

[a] The tolerance for test temperatures shall be ±1.1 C or ±2 F up to 200 F, and ±1 per cent for temperatures above 200 F.

[b] Presoaking shall consist of submerging specimens in water and applying vacuum at 20 in. of mercury until weight equilibrium is reached.

[c] The relative humidity will ordinarily be 95 to 100 per cent.

NOTE.—This property of a system is measured by the depth of penetration of the adhesive into the adherend.

Plasticizer, n.—A material incorporated in an adhesive to increase its flexibility, workability, or distensibility. The addition of the plasticizer may cause a reduction in melt viscosity, lower the temperature of the second-order transition, or lower the elastic modulus of the solidified adhesive.

Retrogradation, n.—A change of starch pastes from low to high consistency on aging.

Shortness, n.—A qualitative term that describes an adhesive that does not string, cotton, or otherwise form filaments or threads during application.

Slippage, n.—The movement of adherends with respect to each other during the bonding process.

Solids Content, n.—The percentage by weight of the non-volatile matter in an adhesive.

NOTE.—The actual percentage of the non-volatile matter in an adhesive will vary considerably according to the analytical procedure that is used. A standard test method must be used to obtain consistent results.

Stringiness, n.—The property of an adhesive that results in the formation of filaments or threads when adhesive transfer surfaces are separated. (See Webbing.)

NOTE.—Transfer surfaces may be rolls, picker plates, stencils, etc.

Tack, n.—The property of an adhesive that enables it to form a bond of measurable strength immediately after adhesive and adherend are brought into contact under low pressure.

Thinner, n.—A volatile liquid added to an adhesive to modify the consistency or other properties. (See also Diluent and Extender.)

Webbing, n.—Filaments or threads that may form when adhesive transfer surfaces are separated. (See Stringiness.)

NOTE.—Transfer surfaces may be rolls, picker plates, stencils, etc.

REVISIONS OF STANDARDS, IMMEDIATE ADOPTION

The committee recommends for immediate adoption revisions as follows of the Standard Method of Test for Resistance of Adhesive Bonds to Chemical Reagents (**D 896 – 51**)[3] and accordingly asks for the necessary nine-tenths affirmative vote at the Annual Meeting in order that the revisions may be referred to letter ballot of the Society.

Section 4.—Add the following new paragraphs:

(*o*) Iso-Octane (2,2,4 trimethylpentane)

(*p*) Hydrocarbon Mixture No. 1

(*1*) Iso-Octane (2,2,4 trimethylpentane)	600 ml
(*2*) Benzene	50 ml
(*3*) Toluene	200 ml
(*4*) Xylene	150 ml

(*q*) Hydrocarbon Mixture No. 2	
(*1*) Iso-Octane (2,2,4 trimethylpentane)	700 ml
(*2*) Toluene	300 ml
(*r*) Standard Jet Fuel No. 1	
(*1*) Toluene	300 ml
(*2*) Cyclohexane	600 ml
(*3*) Iso-Octane (2,2,4 trimethylpentane)	100 ml
(*4*) *n*-Butyl Disulfide	10 ml
(*5*) *n*-Butyl Mercaptan (equivalent to 0.005 weight per cent of mercaptan sulfur)	0.125 g
(*s*) Standard Jet Fuel No. 2	
(*1*) Toluene	300 ml
(*2*) Cyclohexane	600 ml
(*3*) Iso-Octane (2,2,4 trimethylpentane)	100 ml
(*4*) *n*-Butyl Disulfide	1 ml
(*5*) *n*-Butyl Mercaptan (equivalent to 0.0004 weight per cent mercaptan sulfur)	0.010 g

REAPPROVAL OF STANDARDS

The committee recommends the reapproval of the following standards which have stood for more than six years without revision:

Standard Method of Test for:

Tensile Properties of Adhesives (**D 897 – 49**),
Applied Weight per Unit Area of Dried Adhesive Solids (**D 898 – 51**),
Applied Weight per Unit Area of Liquid Adhesive (**D 899 – 51**),
Peel or Stripping Strength of Adhesives (**D 903 – 49**),
Shear Strength of Adhesives by Compression Loading (**D 905 – 49**),
Strength of Adhesives in Plywood Type Construction in Shear by Tension Loading (**D 906 – 49**), and
Cleavage Strength of Metal-to-Metal Adhesives (**D 1062 – 51**).

TENTATIVES CONTINUED WITHOUT REVISION

The committee recommends the continuation without revision of the following tentatives:

Tentative Method of Test for:

Strength of Adhesives in Shear by Tension Loading (Metal-to-Metal) (**D 1002 – 53 T**),
Resistance of Adhesives for Wood to Cyclic Laboratory Aging Conditions (**D 1183 – 55 T**),
Adhesives Relative to Their Use as Electrical Insulation (**D 1304 – 54 T**),
Susceptibility of Dry Adhesive Films to Attack by Roaches (**D 1382 – 55 T**), and
Susceptibility of Dry Adhesive Films to Attack by Laboratory Rats (**D 1383 – 55 T**).

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[4]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Strength Properties (C. E. Britt, chairman) is completing a Recommended Practice for Conducting Creep Testing of Metal-to-Metal Adhesives.

An interlaboratory test program to determine the rate of loading to be specified in the Method of Test for Strength Preparation of Adhesives in Shear by Tension Loading (Metal-to-Metal) (**D 1002 – 53 T**) has been completed and the data statistically outlined. Recommendations on this method may be made in the near future. The climbing drum test procedure has been revised by recent studies and is being prepared for presentation to the committee. The second draft of the T-Peel test procedure is in preparation, as well as a proposed recommended practice for determining the suitability of chambers used to conduct extreme temperature exposures, and tests on adhesive bonded lap-shear specimens.

Subcommittee II on Analytical Tests (G. F. Lipsey, chairman) prepared the three proposed methods of testing hydrogen-ion concentration of dry adhesive films; non-volatile content of phenol, resorcinol, and melamine adhesives; and the filler content of adhesives, referred to earlier in the report.

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[4] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

Methods under preparation include tests for non-volatile content and specific gravity of solvent-type resin adhesives.

Subcommittee III on Tests for Permanency (D. A. George, chairman) is preparing a method for the effect of natural molds on the permanence of adhesives and a recommended practice for the effect of weathering on adhesives while under stress. A study of the effects of nuclear radiation on the strength of adhesive bonds is being considered.

Subcommittee IV on Working Properties (N. J. DeLollis, chairman) is continuing work on the determination of pressure-sensitive tack. Methods for the measurement of flow properties, viscosity, and penetration of adhesives are being prepared.

Subcommittee V on Specifications (S. L. Adams, chairman) prepared for presentation to the Society methods for testing water absorptiveness of paper labels and bonding permanency of water or solvent soluble liquid adhesives for labeling glass bottles, and a specification for bonding permanency of water or solvent soluble liquid adhesives for labeling glass bottles, all referred to earlier in the report.

A specification for adhesives for acoustical materials has been developed for presentation to the committee. Methods under development include tests for the bonding permanency of adhesives for sealing top flaps of fiberboard shipping cases, the selection of fiberboard case samples for the bonding test, and adhesive sampling procedures. Specifications in preparation include adhesives for sealing top flaps of fiberboard shipping containers, general purpose (mending) adhesives, and polyvinyl acetate resin emulsion adhesives (alkali dispersible).

Subcommittee VI on Nomenclature (G. W. Koehn, chairman) prepared the 14 definitions for inclusion in Definitions D 907 referred to earlier in the report.

Subcommittee VII on Research Problems (J. E. Rutzler, Jr., chairman) has reviewed research studies in the adhesives field. Research reports were presented on: bonding with molten polymers, and adhesive rupture before formation of oxide films in adhesive bonds. Problems to be included in "Challenges in Materials Research" have been chosen for preparation by the committee.

Subcommittee X on Wood Adhesives (A. A. Marra, chairman) presented a survey of adhesive testing in the fields of plywood, laminations, and assembly gluing. Representatives of the Forest Products Laboratory and the Hardwood Plywood Inst. reviewed the activities of their organizations with regard to research and control testing of plywoods and assemblies. The subcommittee is preparing a detailed questionnaire to circulate to those in a position to contribute to the development of test methods for wood-to-wood adhesives.

New sections formed in the subcommittee were set up on the basis of glued products which are presumed to demand similar properties in adhesives and thus utilize similar test methods, as follows: Section A on Lumber Adhesives (R. F. Blomquist, chairman), Section B on Veneer Adhesives (John Butler, chairman), Section C on Pulp and Particle Board Binders (A. A. Marra, chairman), Section D on Assembly Adhesives (R. F. Snider, chairman), Section E on Adhesives for Bonding Wood to Other Materials (C. B. Hemming, chairman), and Section F on Special Problems (Ben Jayne, chairman).

Subcommittee XI on Metal Bonding Adhesives (F. H. Bair, acting chairman) has organized four sections as follows: Section A on Mechanical Properties, Section B on Durability, Section C on

Metal Surface Preparation, and Section D on Inspection and Quality Control.

This new subcommittee is currently reviewing existing methods to determine if revisions are necessary. New methods which are being considered for development include evaluation of surface preparation, and procedures which are of particular interest to the aircraft industry.

This report has been submitted to letter ballot of the committee, which consists of 127 voting members; 79 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

R. F. BLOMQUIST,
Chairman.

J. J. LAMB,
Secretary.

REPORT OF COMMITTEE D-15

ON

ENGINE ANTIFREEZES*

Committee D-15 on Engine Antifreezes held one meeting during the year: in Washington, D.C., on March 28, 1958. Meetings of subcommittees were held in Philadelphia, Pa., on November 14 and 15, 1957, and in Washington, D.C., on March 26 and 27, 1958.

There have been two resignations from the committee during the year. Two new members were added to the committee. The committee is composed of 36 members, of whom 10 are classified as producers, 15 as consumers, and 11 as general interest members.

Committee D-15 is now represented on the Advisory Committee on Corrosion.

The officers and members of the Advisory Committee elected for the ensuing term of two years are as follows:

Chairman, R. E. Vogel.

Vice-Chairman, F. G. Church.

Secretary, C. O. Durbin.

Advisory Committee, Members-at-Large, V. O. Hatch and W. D. McMaster.

REVISION OF TENTATIVE

The committee recommends revisions as appended hereto[1] of the Tentative Specification for Hydrometer-Thermometer Field Tester for Engine Antifreezes (**D 1124 - 53 T**)[2] and continuation of the specification as tentative.

ADOPTION OF TENTATIVES AS STANDARD WITHOUT REVISION

The committee recommends that the following tentatives be approved for reference to letter ballot of the Society for adoption as standard without revision:

Tentative Method of Test for:

Ash Content of Concentrated Engine Antifreeze (**D 1119 - 53 T**),[2] and

pH of Concentrated Engine Antifreezes (**D 1287 - 53 T**).[2]

REVISION OF STANDARD, IMMEDIATE ADOPTION

The committee recommends for immediate adoption revisions as follows of the Standard Method of Test for Specific Gravity of Concentrated Engine Antifreezes by the Hydrometer (**D 1122 - 53**),[2] and accordingly asks for the necessary nine-tenths affirmative vote at the Annual Meeting in order that the revisions may be referred to letter ballot of the Society:

Section 3(a).—Delete this section and replace with the following:

> 3. (*a*) *Hydrometers.*—Hydrometers shall be of glass, graduated in specific gravity as listed in Table I, and shall conform to the Tentative Specifications for ASTM Hydrometers (ASTM Designation: E 100). The glass shall have a cubical coefficient of expansion of approximately 0.0000128 per 1 F at 60 F.

New Table.—Add a new Table I as shown in the accompanying Table I.

TENTATIVE CONTINUED WITHOUT REVISION

The committee recommends the continuation without revision of the Tentative Method for Glassware Corrosion Test for Engine Antifreezes (**D 1384 - 55 T**).[2] Modifications of this tentative method are being investigated.

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* Sixty-first Annual Meeting of the Society, June 22–27, 1958.

[1] The revised tentative appears in the 1958 Book of ASTM Standards, Part 8.

[2] 1955 Book of ASTM Standards, Part 5.

TABLE I.—AVAILABLE HYDROMETERS.
(New Table I, Standard Method D 1122 - 53)

ASTM Designation	Type	Specific Gravity Range	
		Total	Each Hydrometer
84H to 88H...	for general use in light liquids	0.745 to 1.005	0.060
111H to 117H.	for general use in heavy liquids	0.995 to 1.355	0.060

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[3]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee II on Antifreeze Field Testers (F. G. Church, chairman) revised the Tentative Specification for the Hydrometer-Thermometer Field Tester for Engine Antifreezes (D 1124 - 53 T), referred to earlier in the report, to include a test method for obtaining basic data with hydrometers for the initial calibration of these testers for primary use by antifreeze producers and tester manufacturers.

Subcommittee III on Physical Properties (R. E. Vogel, chairman) revised the Standard Method of Test for Specific Gravity of Concentrated Engine Antifreezes by the Hydrometer (D 1122 - 53), referred to earlier in the report, to prescribe the use of standard hydrometers meeting ASTM Specification E 100. Consideration is being given to a rapid method for determining melting point of engine antifreeze solutions.

Subcommittee IV on Chemical Properties (J. J. Ondrejcin, chairman) is revising the Method of Test for Water in Concentrated Engine Antifreezes by the Iodine Reagent Method (D 1123 - 54) to replace the methanol base reagent with the glycol ether base reagent used in Method D 1364 - 55 T. A significance of test section is also under investigation. The Study Group on Glycol Analysis is collecting methods to distinguish between various glycols.

Subcommittee VI on Simulated and Actual Service Testing (C. H. Sweatt, chairman) has two active study groups. During the past year, the Study Group on Glassware Test Methods (E. L. Gibson, chairman) completed a second interlaboratory test on a laboratory screening method for foaming tendency. Modifications are under study. The Study Group on Service Simulation Test Methods (V. O. Hatch, acting chairman) is continuing the development of apparatus and operating procedure for a bench-type circulating test that would evaluate corrosion and foaming properties under conditions that simulate service. A prototype assembly is nearly finished. Members of Subcommittee VI have found the results of tests with "standard" specimens provided by the Chemical Specialties Manufacturers Assn. to be equal to earlier results from the standpoint of reproducibility and repeatability.

Subcommittee VII on Specifications (B. E. Tiffany, chairman) is continuing work on a specification for engine antifreeze.

Subcommittee VIII on Editorial and Coordination of Test Methods (G. Kaufman, chairman) was established during the year. This subcommittee has reviewed and advised on the revised methods presented in this report.

This report has been submitted to letter ballot of the committee, which consists of 36 voting members; 28 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

R. E. VOGEL,
Chairman.

C. O. DURBIN,
Secretary.

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[3] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

REPORT OF COMMITTEE D-16
ON
INDUSTRIAL AROMATIC HYDROCARBONS AND RELATED MATERIALS*

Committee D-16 on Industrial Aromatic Hydrocarbons and Related Materials and its subcommittees held two meetings during the year: on June 20 and 21, 1957, in Atlantic City, N. J., and on February 10 and 11, 1958, in St. Louis, Mo.

The officers elected for the ensuing term of two years are as follows:

Chairman, W. E. Sisco.
Vice-Chairman, M. Mitchell.
Secretary, K. H. Ferber.

NEW TENTATIVE

The committee, jointly with Committee D-2, recommends for publication as tentative the Method for Calculation of Volume and Weight of Benzene, Toluene, and Paraxylene, as appended hereto.[1] This is a modification of the proposed method published as information with the 1957 Report of Committee D-16.

TENTATIVE CONTINUED WITHOUT REVISION

The committee recommends for continuation without revision the Tentative Specification for Industrial 90 Benzene (**D 837 – 56 T**).

REAPPROVAL OF STANDARDS

The committee recommends the reapproval of the following sixteen standards which have been published for six years or longer without revision:

Standard Specifications for:

Nitration Grade Benzene (**D 835 – 50**),
Industrial Grade Benzene (**D 836 – 50**),
Refined Solvent Naphtha (**D 838 – 50**),
Crude Light Solvent Naphtha (**D 839 – 50**),
Crude Heavy Solvent Naphtha (**D 840 – 50**),
Industrial Grade Toluene (**D 842 – 50**),
Nitration Grade Xylene (**D 843 – 50**),
Industrial Grade Xylene (**D 844 – 50**),
Five-Degree Xylene (**D 845 – 50**), and
Ten-Degree Xylene (**D 846 – 50**).

Standard Methods of Test for:

Acidity of Benzene, Toluene, Xylenes, Solvent Naphthas, and Similar Industrial Aromatic Hydrocarbons (**D 847 – 47**),
Copper Corrosion of Industrial Aromatic Hydrocarbons (**D 849 – 47**),
Paraffins in Industrial Aromatic Hydrocarbons (**D 851 – 47**),
Solidification Point of Benzene (**D 852 – 47**),
Color and Hydrogen Sulfide and Sulfur Dioxide Content (Qualitative) of Industrial Aromatic Hydrocarbons (**D 853 – 47**), and
Thiophene in Benzene (**D 931 – 50**).

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[2]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee A on Monocyclic Aromatics (S. S. Kurtz, Jr., chairman) with the cooperation of Committee D-2 on Petroleum Products and Lubricants prepared the appended Method for Calculation of Volume and Weight of Benzene,

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* Sixty-first Annual Meeting of the Society, June 22–27, 1958.

[1] The new tentative appears in the 1958 Book of ASTM Standards, Part 8.

[2] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

TABLE I.—SUMMARY OF COOPERATIVE DATA ON BROMINE INDEX, FEB. 1957.

	D-16-17	D-16-21	SOI No.1[a]	D-16-19	D-16-23	D-16-20	D-16-24	SOI No. 2[a]	SOI No. 3[a]	D-16-22	D-16-18	UOP 105-8519[b]
Type of Sample[c]	A	A	B	A	A	A	A	B	B	A	A	B
ppm:												
Cyclohexene	0.0	0.0	...	20.0	20.0	50.0	50.0	...	...	250.0	250.0	...
Thiophene	0.0	200.0	...	0.0	200.0	0.0	200.0	...	...	200.0	0.0	...
Average Results:												
Potentiometric[d] (7 to 10 labs)	0.29	0.87	2.7	3.82	4.41	9.4	9.54	12.9	24.0	44.9	45.3	143
Coulometric[e] (4 labs)	0.66	1.14	4.85	4.26	5.00	9.8	10.8	14.6	26.6	46.0	45.2	147
Difference	+0.37	+0.27	+2.15	+0.44	+0.59	+0.4	+1.26	+1.7	+2.6	+1.1	−0.1	+4
Repeatability, Standard Deviation:												
Potentiometric[d] (7 to 10 labs)	0.23	0.17	0.61	0.49	0.17	0.97	0.35	1.5	3.3	1.5	0.05	8.9
Coulometric[e] (4 labs)	0.09	0.12	0.14	0.29	0.24	0.21	0.26	0.23	0.42	0.56	0.80	5.0
Reproducibility, Standard Deviation:												
Potentiometric[d] (7 to 10 labs)	0.31	0.65	1.17	0.63	1.64	1.25	1.10	3.61	6.7	4.1	2.43	17.3
Coulometric[e] (4 labs)	0.16	0.06	0.56	0.05	0.11	0.30	0.50	1.17	5.2	0.58	0.72	15.0

[a] SOI Nos. 1, 2, and 3 are commercial benzene samples.

[b] UOP 105-8519 is a plant stream before treatment to reduce bromine index.

[c] A = Known blend of cyclohexene or thiophene in benzene; B = special samples from cooperators.

[d] In accordance with Tentative Method of Test for Bromine Index of Aromatic Hydrocarbons by Potentiometric Titration (D 1491 – 57 T).

[e] In accordance with Tentative Method of Test for Bromine Index of Aromatic Hydrocarbons by Coulometric Titration (D 1492 – 57 T).

Toluene, and Paraxylene.[1] The subcommittee is working on the revision of Specifications for Industrial 90 Benzene (D 837 - 56 T), Nitration Grade Toluene (D 841 - 50), and Methods for Acid Wash Color of Benzene, Toluene, Xylenes, Refined Solvent Naphthas, and Similar Industrial Aromatic Hydrocarbons (D 848 - 47), and Specific Gravity of Industrial Aromatic Hydrocarbons (D 891 - 51).

The summary of cooperative data on the Tentative Methods of Test for Bromine Index of Aromatic Hydrocarbons by Potentiometric Titration (D 1491 - 57 T), and Bromine Index by Coulometric Titration (D 1492 - 57 T) is set forth in the accompanying Table I.

Subcommittee B on Polycyclic Aromatics (M. Mitchell, chairman) expects to include solidification point, evaporation residue and water content in a specification to cover crude naphthalene.

Subcommittee C on Phenolic Compounds (D. F. Pontz, chairman) has found that the conventional Karl Fischer method for determination of water content is not suitable for phenol and a suitable modification has been developed. Investigation is under way for methods for determining color and water solubility of refined phenol.

Subcommittee D on Nitrogen Heterocyclics (W. E. Sisco, chairman) is investigating the use of the solidification point method as a means of determining the purity of refined quinoline.

Subcommittee E on Thermometry (K. H. Ferber, chairman) has submitted samples of phenol, naphthalene, and phthalic anhydride to the National Bureau of Standards for testing in sealed glass cells. The cells, provided with a thermometer well, are for use in standardizing solidification point thermometers.

Subcommittee F on Editorial and Nomenclature (E. T. Scafe, chairman) is functioning as a consultant body to aid in the programs of the other subcommittees.

Subcommittee G on Statistical Procedure (W. J. Tancig, chairman) has prepared a proposed method on general statistical practices for use of the other subcommittees.

This report has been submitted to letter ballot of the committee, which consists of 55 voting members; 47 members returned their ballots, of whom 46 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

D. F. GOULD,
Chairman.

F. J. POWELL,
Secretary.

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee D-16 presented to the Society through the Administrative Committee on Standards revisions of the Tentative Method of Test for Solidification Points of Chemicals (D 1493 - 57 T). The revisions were accepted by the Standards Committee on September 9, 1958, and the revised tentative method appears in the 1958 Book of ASTM Standards, Part 8.

REPORT OF COMMITTEE D-17
ON
NAVAL STORES*

Committee D-17 on Naval Stores held one meeting during the year: on June 20, 1957, at Atlantic City, N. J., in connection with the Annual Meeting of the Society.

The committee reports the loss through death on June 13, 1957, of Robert C. Palmer, Vice President and Chemical Director of Newport Industries, Inc., one of its long-time collaborating company representatives. Another member, G. R. Tennent, Chief Chemist of the Hummel Ross Fiber Division, Continental Can Co., was lost through retirement.

The special committee working on revisions of Federal Specifications for naval stores, on which the Society is represented by the chairman of Committee D-17, has completed the revisions and the specifications are now available in their revised form. The specifications include Turpentine (TT-T-801a), Rosin (LLL-R-626b), and Dipentene (TT-D-376c.)

The officers elected for the ensuing term of two years are as follows:

Chairman, S. R. Snider.
Vice-Chairman, J. L. Boyer.
Secretary, W. A. Kirklin.

NEW TENTATIVE

The committee recommends for publication as tentative the Methods of Test for Fatty Acids Content of Tall Oil Rosin as appended hereto.[1]

ADOPTION OF TENTATIVE AS STANDARD WITHOUT REVISION

The committee recommends that the Tentative Methods of Testing Tall Oil (**D 803 – 55 T**)[2] be approved for reference to letter ballot of the Society for adoption as standard without revision.

REVISION OF STANDARDS, IMMEDIATE ADOPTION

The committee recommends for immediate adoption revisions of the following standards as indicated, and accordingly requests the necessary nine-tenths affirmative vote at the Annual Meeting in order that the revisions may be referred to letter ballot of the Society.

Standard Methods of Sampling and Testing Turpentine (D 233 – 57):[3] Revise Sections 13 and 14 as appended hereto.[4]

The proposed revision is dictated by inaccuracies and disagreements that frequently arise in the results obtained by testing laboratories working on turpentine or related material, which in many cases have been brought about by improper carrying out of the test procedure.

Standard Method of Test for Volatile Oil in Rosin (D 889 – 50):[2]

Section 4.—Renumber and change to read as follows:

5. *Additional Examination of Oil.*—Drain off

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* Sixty-first Annual Meeting of the Society, June 22–27, 1958.

[1] The new tentative appears in the 1958 Book of ASTM Standards, Part 8.

[2] 1955 Book of ASTM Standards, Part 4.

[3] 1957 Supplement to Book of ASTM Standards, Part 4.

[4] The revised standard appears in the 1958 Book of ASTM Standards, Part 8.

the water, transfer the oil to a small glass container, stopper, and allow to settle until clear. Note the odor and taste and determine the refractive index. For further information on the nature of the oil, volume permitting, make a polymerization test according to the procedure given in Standard Methods of Sampling and Testing Turpentine (ASTM Designation: D 233).

Section 5.—Renumber and change to read as follows:

4. *Report.*—Properly made rosin yields not more than 0.5 ml. of residual volatile oil per 50 g. of sample. If the recovered oil is within such limit, report the rosin as free from excess volatile oil. If more than 0.5 ml. of oil is recovered, report the total volume obtained.

NOTE.—The volatile oil recovered from gum rosin consists primarily of high-boiling terpene components of the crude gum, most of which under proper distillation will have been recovered as turpentine by the time the rosin is drawn from the still. Faulty distillation or premature withdrawal may yield a rosin giving a higher than normal recovery of oil, with a refractive index below 1.483. The polymerization residue should have a refractive index not lower than 1.500. In the case of wood rosins, generally obtained by extraction from stumpwood with a petroleum or naphtha solvent, any recovered oil would probably consist of unrecovered solvent. The refractive index of both the recovered oil and the polymerization residue therefrom would be well under the above figures for gum rosin.

Standard Method of Test for Water in Liquid Naval Stores (D 890 - 51):[2]

Section 3.—Change the Note to read as follows:

NOTE.—*Alternate Titration*—The end point may be determined electrometrically by means of the apparatus and procedure described in Section 3a of Tentative Method of Test for Water in Lacquer Solvents and Diluents (ASTM Designation: D 1364).

Sections 4(a) and 5(a).—Add to the second sentence "or alternately to an electrometric end point."

Standard Method of Test for Iron in Rosin (D 1064 - 51):[2]

Section 4(b).—Change to read as follows:

(*b*) *Dishes*, Vycor, silica, or porcelain, 50- and 100-ml capacity.

NOTE.—Platinum or platinum-rhodium dishes are not recommended as they sometimes cause a color interference with the phenanthroline reagent.

Section 7.—In the first sentence, change "a platinum or Vycor dish" to read "a Vycor, silica, or porcelain dish."

EDITORIAL CHANGE

The committee recommends editorial changes as follows in the Standard Methods of Sampling and Grading Rosin (**D 509 - 55**):[2]

Section 3.—Change the explanatory Note to read as follows:

NOTE.—Gum rosin, from the oleoresin or "gum" obtained from living pine trees, is made by distilling individual batches or charges of the gum, the size of which depends on the capacity of the still. At the end of the distillation, the rosin is drawn either into steel drums having a tare weight of 17 lb, and uniformly filled to contain 517 net lb of rosin, or into multi-wall paper bags containing 100 net lb. A batch or charge may consist of from 8 to 15 drums, or the equivalent number of bags. Two samples of the hot molten rosin are taken from each batch, from the first and last filled containers, and poured into molds for grading. The color of the rosin may vary slightly between subsequent batches or between the first and last container filled from a batch. After cooling, the drums and bags are often transferred to a storage yard or shed to await shipment. Consequently, in every carload shipment, the drums or bags will represent a number of batches and inspections. Shipments of drum rosin moving in commerce from a central storage point usually represent the output of more than one producer. The rosin may have been made from gum collected at different seasons of the year. It will be seen from the above that a correct appraisal of the over-all grade of a commercial lot of rosin cannot be obtained from just a limited number of samples.

REAPPROVAL OF STANDARDS

The committee recommends the reapproval of the following standards:

Standard Method of:

Test for Toluene Insoluble Solid Matter in Rosin

(Chiefly Sand, Chips, Dirt, and Bark) (**D 269 - 52**),
Sampling and Testing Pine Tars and Pine-Tar Oils (**D 856 - 49**),
Test for Ash in Rosin (**D 1063 - 51**), and
Testing Rosin Oils (**D 1131 - 53**).

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[5]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Softening Point of Rosin (J. M. Schantz, chairman) is studying the possibilities of standardizing a method of test for softening point of rosin known as the thermometer drop method. Among the tests having the widest application is one developed by Hercules Powder Co. (see page 470, ASTM *Proceedings*, 1948).

Many manufacturers of wood rosin use some form of drop method as a rapid plant control test in their regular production operations. Several consumers of rosin have also found such a method useful and have expressed an interest in further study of such a test procedure. This method requires the use of a special thermometer having a bulb with closely specified dimensions, as well as skill in the technique of molding a prescribed weight of the rosin sample onto the thermometer bulb. The rest of the apparatus used in this test is of a type usually found in general testing laboratories.

Subcommittee II on Crystallization (S. R. Snider, chairman) has prepared a report covering the relative tendency to crystallize from solution for 46 gum rosins specially selected from a larger previously tested lot which is representative of the output of the entire gum naval stores industry and production throughout the regular operating seasons. The report will show comparisons between the results obtained on these samples using the method published as information in the Appendix to the 1955 Annual Report of the committee,[6] the results obtained with the so-called Bakelite method, and a method published by Palkin and Smith in the Journal of the American Oil Chemists Society (Vol. 15, 1938, p. 120). This report will be presented for discussion at the forthcoming meeting of Committee D-17.

Subcommittee IV on Chemical Analysis of Rosin (R. Herrlinger, chairman) has conducted an active collaborative analytical study on specially prepared samples of tall oil rosin of an improved method for determining the fatty acid content of tall oil rosin. The importance of such test is indicated by the fact that a tall oil rosin that meets the Federal standards and trade specifications for this class must contain not more than 5 per cent residual fatty acids. Two alternate procedures for determining the resin acid content of tall oil rosin—the Wolfe and the Linder-Persson methods—gave such good agreement that both procedures have been included in the proposed tentative appended hereto.[1] Additional collaborative work is planned on the Linder-Persson method during the coming year. Another project is the preparation of an automatic potentiometric titration procedure for use in the methods of test for acid number and saponification number of rosin.

Subcommittee V on Tall Oil (A. Pollak, chairman) has participated in the work of several subcommittees of Committee D-1, with regard to methods for determining rosin and rosin acids in coating materials or in the tall oil fatty acids used in the manufacture of industrial protective coatings.

Subcommittee VII on Terpene Hydro-

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[5] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

[6] *Proceedings*, Am. Soc. Testing Mats., Vol. 55, p. 531 (1955).

carbons (R. E. Price, chairman) has prepared a revision of the directions for carrying out the several steps in the technique of making the so-called polymerization test on turpentine and other terpene oils. The results of this study are covered by a recommendation for revision of the turpentine test methods referred to earlier in this report.

Subcommittee IX on Definitions (J. L. Boyer, chairman) has prepared and submitted for study three new definitions of terms relating to naval stores, namely, refined tall oil, tall oil fatty acids, and rectified turpentine. These definitions are being considered for inclusion in the Standard Definition of Terms Related to Naval Stores (ASTM D 804). The standard definition for sulfate pine oil is also under consideration for revision in view of recent technical developments in its manufacture.

This report has been submitted to letter ballot of the committee, which consists of 34 members; 22 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

V. E. GROTLISCH,
Chairman.

W. A. KIRKLIN,
Secretary.

REPORT OF COMMITTEE D-18
ON
SOILS FOR ENGINEERING PURPOSES*

Committee D-18 on Soils for Engineering Purposes held its Annual Meeting in Atlantic City, N. J., on June 19, 1957. A committee meeting was also held on December 10, 1957, at Mexico City during the Joint Meeting of Committee D-18 and the Sociedad Mexicana de Mecanica de Suelos. Executive Committee Meetings were held on June 17, 1957, at Atlantic City and on January 5, 1958, at Washington, D.C.

A Soils Session, consisting of seven papers, was held during the 1957 Annual Meeting of the Society in Atlantic City. These have been published in the 1957 *Proceedings* of the Society. Five Soils Sessions, consisting of 15 papers, were held during the Joint Meeting in Mexico City. These papers and related discussions have been published in both Spanish and English in a separate publication by the Society.[1]

Hogentogler Award.—The Special Committee for the Hogentogler Award recommended that the award be given to Professor D. M. Burmister for his paper entitled "Application of Environmental Testing of Soils."[2]

The revision of "Procedures for Testing Soils" has been completed by the Special Committee on Revision of Procedures for Testing Soils, and the revised procedures are being published by the Society.

Committee D-18 now consists of a total of 137 individual members and 19 consultants. There are 107 voting members, of whom 20 are classified as producers, 38 as consumers, and 49 as general interest.

The officers elected for the ensuing term of two years are as follows:

Chairman, E. J. Kilcawley.
First Vice-Chairman, W. S. Housel.
Second Vice-Chairman, F. J. Converse.
Secretary, W. G. Holtz.
Assistant Secretary, A. A. Wagner.

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1957 Annual Meeting, Committee D-18 presented to the Society through the Administrative Committee on Standards the recommendation that the Tentative Method of Test for Load-Settlement Relationship of Individual Piles Under Vertical Axial Load (**D 1143 – 50 T**) be revised. This recommendation was accepted by the Standards Committee on December 2, 1957, and the revised tentative appears in the 1957 Supplement to Book of ASTM Standards, Part 3.

NEW TENTATIVES

The committee recommends for publication as tentative the following methods as appended hereto or otherwise indicated:[3]

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* Sixty-first Annual Meeting of the Society, June 22–27, 1958.

[1] ASTM Special Technical Publication, *ASTM STP No. 232.*

[2] *Proceedings*, Am. Soc. Testing Mats., Vol. 56, p. 1351 (1956).

[3] The new and revised tentatives appear in the 1958 Book of ASTM Standards, Part 4.

Tentative Method of Test for:

Moisture-Penetration Resistance Relations of Fine-Grained Soil (Jointly with Committee D-4 on Road and Paving Materials),
Density of Soil in Place by the Sand Cone Method (Jointly with Committee D-4 on Road and Paving Materials), and
Determination of the Moisture-Density Relations of Soils Using a 10-lb Rammer and 18-in. Drop (Jointly with Committee D-4 on Road and Paving Materials).

Tentative Method for:

Thin-Walled Tube Sampling of Soils,[4] and
Penetration Test and Split-Barrel Sampling of Soils.[5]

REVISIONS OF TENTATIVE

The committee recommends revisions of the Tentative Method of Test for Moisture-Density Relations of Soils (**D 698 – 57 T**)[4] as indicated in the 1958 Report of Committee D-4[3] and continuation of the method as tentative.

REVISIONS OF STANDARDS, IMMEDIATE ADOPTION

The committee recommends for immediate adoption revisions as indicated of the following standards, and accordingly asks for the necessary nine-tenths affirmative vote at the Annual Meeting in order that the revisions may be referred to letter ballot of the Society:

Standard Method of Preparing Soil Samples for Grain-Size Analysis and Determination of Subgrade Soil Constants (D 421 – 39):[5]

Title.—Revise the title to read "Standard Method of Dry Preparation of Soil Samples for Grain-Size Analysis and Determination of Soil Constants."

Section 1.—Insert the word "dry" before "preparation" and delete the word "subgrade."

Section 2 (c).—Delete the No. 200 sieve in the tabulation in this section.

Section 3.—In Paragraph (*a*) replace the word "mechanical" with the word "grain-size."

In Paragraph (*b*) revise the first four lines to read: "(*b*) *Tests for Soil Constants.* For the tests for the soil constants, material passing the No. 40 sieve is required in total amount of 270 g, allocated as follows:"

Section 6.—Delete the word "subgrade" in the title and in the last line of this section.

Standard Method of Test for Specific Gravity of Soils (D 854 – 52):[5]

Section 3 (a).—In line 4 revise "of 50 ml" to read "of at least 50 ml."

Section 5.—In Paragraph (*b*) change the last sentence to read as follows:

> Samples of clay soils containing their natural moisture content shall be dispersed in distilled water before placing in the flask, using the dispersing equipment specified in Method D 422.

New Note.—Add a new Note 2 following Paragraph (*c*) to read as follows, numbering the present note as Note 1:

> NOTE 2.—The minimum volume of slurry that can be prepared by the dispersing equipment specified in Method D 422 is such that a 500-ml flask is needed as the pycnometer.

REAPPROVAL OF STANDARD

The committee recommends the reapproval of the Standard Methods of Surveying and Sampling Soils for Highway Subgrades (**D 420 – 50**) which has stood for more than six years without revision.

TENTATIVES CONTINUED WITHOUT REVISION

The committee recommends the continuation without revision of the following standards:

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[4] 1957 Supplement to Book of ASTM Standards, Part 3.

[5] 1955 Book of ASTM Standards, Part 3.

Tentative Methods of:

Grain-Size Analysis of Soils (**D 422 – 54 T**),
Testing Soil-Bituminous Mixtures (**D 915 – 47 T**), and
Soil Investigation and Sampling by Auger Boring (**D 1452 – 56 T**).

Tentative Method of Test for:

Liquid Limit of Soils (**D 423 – 54 T**),
Plastic Limit and Plasticity Index of Soils (**D 424 – 54 T**),
Shear Strength of Flexible Road Surfaces, Subgrades, and Fills by the Burggraf Shear Apparatus (**D 916 – 47 T**), and
Water-Soluble Chlorides Present as Admixes in Graded Aggregate Road Mixes (**D 1411 – 56 T**).

Tentative Specifications for:

Materials for Soil-Aggregate Subbase, Base, and Surface Courses (**D 1241 – 55 T**).

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[6]

ACTIVITIES OF SUBCOMMITTEES

Special Committee on Revision of Procedures for Soil Testing (W. J. Turnbull, chairman).—This special committee, which consists of the chairmen of the R Subcommittees as members, and Bonner S. Coffman as secretary, held a meeting on June 17 to review the introductions for the several parts of the procedures and other material submitted for the procedures. The special committee completed its work for this revision during the year.

Special Committee for Securing Papers and Arrangements for the Mexico City Meeting (W. G. Holtz, chairman, and E. A. Abdun-Nur and T. W. Van Zelst, members).—Arrangements were made and 15 papers secured from U. S. and Mexican engineers for the Joint Meeting of ASTM Committee D-18 and the Sociedad Mexicana de Mecanica de Suelos, which was held during the week of December 8, 1957, at the University of Mexico. The committee assisted in the review of papers and discussions, and has completed its work relative to the publication of these papers and discussions by the Society.[1]

Subcommittee G-1, Editorial (E. E. Bauer, chairman) reviewed the proposed new tentatives and revisions of tentatives and standards referred to earlier in the report as well as those accepted by the Administrative Committee on Standards since the 1957 Annual Meeting of the Society.

In addition, the chairman has been reviewing editorially the items to be published in the Third Edition of "Procedures for Testing Soils."

Subcommittee G-2 on Special Papers (H. Bolton Seed, chairman) has arranged for the presentation of 10 papers for a 2-session symposium on the subject of Soil Mechanics in Highway Design and Construction, and 5 papers for a General Soils Session on miscellaneous subjects for the 1958 Annual Meeting.

Subcommittee G-3 on Nomenclature and Definitions (C. R. Foster, chairman).—In connection with the revision of definitions in the draft of Standard Definitions of Terms and Symbols Relating to Soil Mechanics (D 653 – 42 T), a second joint meeting of the ASCE Committee and Subcommittee G-3 was held during the year. Revisions agreed upon have been incorporated and the final glossary has been reproduced and distributed to members of Committee D-18 for letter ballot approval. It is recommended that the revised tentative standard replace Definitions D 653 – 42 T.

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[6] The letter ballot vote on these recommendations was favorable; the results of the ballot are on record at ASTM Headquarters.

Subcommittee R-1 on Surface and Subsurface Reconnaissance (L. E. Gregg, chairman).—The subcommittee continued its work in task groups on the preparation of recommended procedures for reconnaissance techniques. One meeting for reports and discussion was held during the year.

Subcommittee R-2 on Sampling and Related Field Testing for Soil Investigations (John P. Gnaedinger, chairman) held a meeting on June 18, 1957, in Atlantic City, N. J. Mr. Gnaedinger was appointed chairman to succeed Jorj Osterberg who resigned.

The subcommittee will consider revisions of Methods D 420 and D 1452 at the Annual Meeting in 1958. The two proposed tentative methods, for Penetration Tests and Split-Barrel Sampling of Soils and for Thin-Walled Tube Sampling of Soils, referred to earlier in the report, were submitted by Subcommittee R-2.

The subcommittee is also preparing a method for core boring and a method for vane shear testing. It is hoped that some action can be taken on these two methods at the meeting of the subcommittee in June 1958.

Subcommittee R-3 on Physical Characteristics of Soils (E. E. Bauer, chairman) recommended revisions of Standard Methods D 421 and D 854. The subcommittee revised the Tentative Method of Test for Moisture-Density Relations of Soils (D 698 - 57 T) and recommended for publication the proposed Tentative Method of Test for Moisture-Penetration Resistance Relations of Fine-Grained Soils and the proposed Tentative Method of Test for Moisture-Density Relations of Soils Using a 10-lb Rammer and an 18-in. Drop.

The cooperative research program to determine the best methods of test for maximum and minimum densities of granular soils is continuing in Section D.

Work is continuing on a proposed Tentative Method of Wet Preparation of Soil Samples for Grain-Size Analysis and Determination of Soil Constants.

Subcommittee R-4 on Physical Properties of Soils (Norman W. McLeod, chairman).—Subcommittee R-4 is primarily concerned with the capillary and permeability characteristics of soils. Since ASTM does not have standard methods for measuring either of these soil properties, Subcommittee R-4 is presently endeavoring to develop test procedures for measuring the permeability of coarser textured soils, and the capillarity of sands and fine textured soils, that can be offered for consideration as standard tests.

Subcommittee R-5 on Structural Properties of Soils (D. M. Burmister, chairman).—The activities of this subcommittee for the past year were confined to a review and study of the suggested methods of test under this subcommittee's jurisdiction in the 1950 Procedures for Testing Soils, and to making recommendations for fourteen suggested methods in the 1958 revision.

It is proposed to write methods of tests for consolidation, direct shear, and triaxial testing for the coming year, covering both apparatus and load applying machines, considering the range of presently used types of apparatus without limiting a test procedure to a single type or size of apparatus.

Subcommittee R-7 on Identification and Classification of Soils (A. A. Wagner, chairman).—The subcommittee reviewed and approved four suggested methods for classification of soils which will appear in the Revised Edition of Procedures for Testing Soils.

Initial steps will be taken during the coming year toward the adoption of

ASTM standards for the classification of soils.

Subcommittee R-8 on Special and Construction Control Tests (H. F. Clemmer, chairman).—The subcommittee held one meeting during the year.

Section A on Base Courses and Subgrades for Highways and Airports has done considerable work on the California Bearing Ratio method of test for bearing of soils. It is hoped to have this method ready for presentation at the next meeting. In addition, the section has under consideration other methods of determining the bearing values of soils.

Section B on Earth Dams and Embankments is considering a method of test for density-in-place by the rubber balloon method and expects to have it ready for presentation soon.

Section C-1 on Cements has approved the following proposed methods of test: (*1*) Making and Curing Soil-Cement Compression and Flexure Test Specimens in the Laboratory, (*2*) Flexure Strength of Soil-Cement Using a Simple Beam with Third Point Loading, (*3*) Compression Strength of Soil-Cement Using Portions of Beams Broken in Flexure (Modified Cube Method), and (*4*) Compression Strength of Molded Soil-Cement Cylinders.

Subcommittee R-9 on Dynamic Properties of Soils (R. K. Bernhard, chairman).—The bibliography appended to the 1953 Symposium on "Dynamic Testing of Soils" is being kept up to date by sending the titles of any old or new publications on Soil Dynamics not included in this bibliography to the chairman of Subcommittee R-9.

A second draft on Definition of Terms Referring to Soil Dynamics was revised according to suggestions made to the subcommittee. It is intended to discuss this draft during the Annual Meeting in June 1958 at Boston, and its submission as a proposed tentative standard.

Reports on some research projects currently under way may be presented at the next meeting of Subcommittee R-9 in June 1958.

Subcommittee R-11 on Pile Load-Bearing Tests (W. S. Housel, chairman).—Subcommittee R-11 completed the revision of Method D 1143. Further study of the method is in progress, covering possible changes on which action was not completed this year. Three other tentative methods are under consideration for tests on batter pile frames, lateral load on single vertical piles or anchors, and a pull test of single vertical piles. Increasing interest in the work of the committee has been expressed by new members of Committee D-18 and several new members have been added.

Joint D-4 and D-18 Subcommittee on Tests and Specifications for Stabilized Soils (H. F. Clemmer, chairman).—The Joint Subcommittee submitted the following proposed new tentatives: Proposed Method of Test for Determining the Density of Soil in Place by the Sand Cone Method; Proposed Method of Test for Moisture Density Relations of Soils Using a 10-lb Rammer and an 18-in. Drop; and Proposed Method of Test for Moisture-Penetration Resistance Relations of Fine-Grained Soils. The Tentative Method of Test for Moisture-Density Relations of Soils (D 698 – 57 T) has been revised. The joint subcommittee recommends that the Tentative Methods D 915, D 916, and D 1411, and Tentative Specifications D 1241 remain as tentative pending further consideration for revision.

The subcommittee is balloting on the four soil-cement test methods referred to earlier in the report under Subcommittee C-1.

This report has been submitted to letter ballot of the committee, which consists of 106 voting members; 78 members returned their ballots, of whom 75 have voted affirmatively and 1 negatively.

Respectfully submitted on behalf of the committee,

E. J. KILCAWLEY,
Chairman.

W. G. HOLTZ,
Secretary.

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee D-18 presented to the Society through the Administrative Committee on Standards revisions of the Tentative Definitions of Terms and Symbols Relating to Soil Mechanics (D 653 – 42 T). The revisions were accepted by the Standards Committee on September 9, 1958, and the revised tentative definitions appear in the 1958 Book of ASTM Standards, Part 4.

REPORT OF COMMITTEE D-19
ON
INDUSTRIAL WATER*

Committee D-19 on Industrial Water and its subcommittees held meetings on June 19 to 21, 1957, in Atlantic City, N. J., and on January 13 to 15, 1958, in New Orleans, La.

A Symposium on the Determination of Dissolved Oxygen in Water,[1] sponsored by Committee D-19, was presented on June 20, 1957, in Atlantic City, N. J., during the Annual Meeting of the Society. The Symposium on Industrial Water and Industrial Waste Water, sponsored by the committee and presented at the Second Pacific Area National Meeting, September, 1956, was published in 1957 as *ASTM STP No. 209*.

A Symposium on Radioactivity in Industrial Water and Industrial Waste Water, sponsored by Committee D-19, will be presented on June 26, 1958, in Boston, Mass., during the Annual Meeting of the Society. The following papers are to be included:

"Radioactivity and Purity Control of APPR Primary Water," by A. L. Medin, Alco Products, Inc.

"Radioactive Waste Processing and Control at the Shippingport Atomic Power Station," by S. F. Whirl and J. A. Tash, Duquesne Light Co.

"Test Methods for Radiation Hazards in Industrial Water," by C. J. Munter, Hagan Chemicals and Controls, Inc.

"Analysis for Radionuclides in Aqueous Wastes from an 'Atomic' Plant," by B. Kahn, Massachusetts Institute of Technology; D. W. Moeller, U. S. Public Health Service; and S. A. Reynolds and J. H. Handley, Oak Ridge National Laboratory.

"The Analysis of Environmental Samples for Radionuclides," by D. L. Reid, General Electric Co.

Two symposia, to be sponsored by the committee, are in preparation for presentation in 1959: "Symposium on Water-Formed Deposits," for the Annual Meeting of the Society in June; and "Symposium on Technical Developments in the Handling and Utilization of Water and Industrial Waste Water," for the Third Pacific Area National Meeting in October.

The Manual on Industrial Water, Fourth Printing of the First Edition (*ASTM STP No. 148-C*) was published in 1958. This printing includes all tentatives and standards under the jurisdiction of the committee accepted by the Society through September, 1957.

The Society's representatives to the Joint Committee on Uniformity of Methods for Water Examination (JCUMWE) submitted various methods to that committee. That committee will submit recommendations to the Society for securing uniformity with methods published by other organizations.

The committee regrets the death of two of its valued members: Frank N. Alquist, of the Dow Chemical Co., who served as chairman of Subcommittee VI on Corrosivity and Performance Testing; and L. C. Flickenger, of Youngstown Sheet and Tube Co., who served as a member of various task groups and as chairman of one.

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* Sixty-first Annual Meeting of the Society, June 22–27, 1958.

[1] Issued as separate publication *ASTM STP No. 219.*

The 1958 Max Hecht Award citation and certificate will be presented on June 26 to Dr. Frederick R. Owens.

The committee consists of 134 members. There are 7 subcommittees, with 63 task groups among them. The task groups are making studies on 25 projects that, when completed, will be offered as proposed methods for acceptance as tentative, and 36 projects for proposed revisions of published tentatives and standards of the committee.

The officers elected for the ensuing term of two years are as follows:

Chairman, Max Hecht.
Vice-Chairman, Frank E. Clarke.
Vice-Chairman, F. R. Owens.
Secretary, O. M. Elliott.

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1957 Annual Meeting, Committee D-19 presented to the Society through the Administrative Committee on Standards the recommendations for revisions of Tentative Methods of Test for Iron in High Purity Water (D 1497 - 57 T) and for acceptance as tentative of the Proposed Method of Test for Oxidation and Reduction Potential of Industrial Water and Industrial Waste Water (D 1498 - 57 T). These recommendations were accepted by the Standards Committee on September 13, 1957, and the methods appear in the 1957 Supplement to Book of ASTM Standards, Part 7.

NEW TENTATIVES

The committee recommends for publication as tentative the following methods as appended hereto:[2]

Methods of Test for:

Dissolved and Gaseous Hydrogen in Industrial Water,
Dissolved Oxygen in Industrial Waste Water,
Effect of a Water in Tubular Heat Exchangers, and
Surface Tension of Industrial Water and Industrial Waste Water.

REVISIONS OF TENTATIVES

The committee recommends revisions as indicated of the following tentatives and their continuation as tentative:

Methods of Test for Phosphate in Industrial Water (D 515 - 55 T):[3] Revise as appended hereto.[2]

Method of Identification of Types of Microorganisms in Industrial Water (D 1128 - 50 T):[3] Revise as appended hereto.[2]

Method of Test for Chemical Oxygen Demand (Dichromate Oxygen Demand) of Industrial Waste Water (D 1252 - 55 T):[3]

Section 6.—Rearrange all reagents alphabetically after changing the title of Paragraphs (*a*) and (*c*) as indicated below.

Paragraph (*a*).—Revise the title from "*Standard Potassium Dichromate Solution* (*0.25 N*)" to read "*Potassium Dichromate, Standard Solution* (*0.25 N*)."

Paragraph (*c*).—Change the title from "*Standard Ferrous Ammonium Sulfate Solution* (*0.25 N*)" to read "*Ferrous Ammonium Sulfate, Standard Solution* (*0.25 N*)."

Section 8 (*a*).—Delete the first two sentences and replace with the following:

> 8. (*a*) To 25.0 ml of 0.2500 *N* $K_2Cr_2O_7$ solution and the necessary dilution water add cautiously 75 ml of H_2SO_4. Cool to room temperature or lower. To the cooled mixture add the sample with thorough mixing (Note 2).

New Note.—Add a new Note 2 to read as follows, renumbering the present Note 2 and subsequent notes accordingly:

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[2] The new and revised tentatives and standards appear in the 1958 Book of ASTM Standards, Part 10.

[3] 1955 Book of ASTM Standards, Part 7.

NOTE 2.—If the sample size is less than 50 ml, it is advantageous to add the necessary amount of dilution water to the $K_2Cr_2O_7$ solution before the addition of the H_2SO_4 rather than diluting the sample to 50 ml. The sample is added to the cooled mixture to minimize the loss of volatile compounds that may be present.

Tentative Methods of Test for Odor of Industrial Waste Water (D 1292 - 53 T):[3]

Table II.—From the list of examples of Sulfury Odors delete the following: "kraft paper, rayon, and cellophane mill wastes," "oil refineries, coffee roasters," "rubber curing chemicals," and "odors around synthetic rubber plants." From the list of Unsaturated Odors delete "slaughter houses."

Method of Test for Hydrazine in Industrial Water (D 1385 - 57 T):[4] Revise as appended hereto.[2]

ADOPTION OF TENTATIVES AS STANDARD WITHOUT REVISION

The committee recommends that the following tentatives be approved for reference to letter ballot of the Society for adoption as standard without revision:

Tentative Methods of Test for:

Suspended and Dissolved Matter (Suspended and Dissolved Solids) in Industrial Water and Industrial Waste Water (**D 1069 - 54 T**),
pH of Industrial Water and Industrial Waste Water (**D 1293 - 55 T**),
Ammonia in Industrial Water and Industrial Waste Water (**D 1426 - 56 T**), and
Residual Chlorine in Industrial Waste Water (**D 1427 - 56 T**).

REVISIONS OF STANDARDS, IMMEDIATE ADOPTION

The committee recommends for immediate adoption revisions as indicated of the following methods and accordingly asks for the necessary nine-tenths affirmative vote at the Annual Meeting in order that the revisions may be referred to letter ballot of the Society:

Standard Method of Reporting Results of Analysis of Industrial Water and Industrial Waste Water (D 596 - 55):[3]

Section 10 (b).—In the table of conversion factors, in the column headed "ppm to epm," revise the following factors to read as indicated:

Ion	ppm to epm
Pb^{++}	0.00965
Fe^{++}	0.03581
Fe^{+++}	0.05372
CO_3^{++}	0.03333
HSO_3^-	0.01233
S^{--}	0.06237
I^-	0.00788
NO_2^-	0.02174

Revise the footnote at the end of the table to read, "Based on International Atomic Weights, 1955."

Standard Method of Test for Iron Bacteria in Industrial Water and Water-Formed Deposits (D 932 - 51):[3]

Section 6 (a).—Revise to read "6. (*a*) *Hydrochloric Acid* (*1:4*).—Mix one volume of HCl (sp gr 1.19) with four volumes of water."

Section 7.—Delete the entire section and replace with the following:

7. (*a*) Samples shall be collected in accordance with the applicable method of the American Society for Testing Materials, as follows:

D 510—Sampling Industrial Water,
D 887—Field Sampling of Water-Formed Deposits.

(*b*) A 1-pt sample of water should be obtained, using a sterile 1-qt bottle. The bottle should not be more than half-filled because of the oxygen demand of suspended matter which may cause the sample to become anaerobic.

(*c*) Deposit or mud samples should be transferred to a wide-mouth bottle and clear chlorine-free water added to cover the deposit.

NOTE.—In mud samples, viable organisms, when present, are usually found at the mud-water interface.

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[4] 1957 Supplement to Book of ASTM Standards, Part 7.

(*d*) The samples should be protected from sunlight and preferably preserved at not over 75 F during transportation.

Section 9.—Delete the present paragraph and replace with the following:

9. The report should state "Present" or "Not found, probably absent." A statement should be made as to the relative abundance of organisms present. A negative report should be made only after examination of several slides.

Methods of Test for Sulfate-Reducing Bacteria in Industrial Water and Water-Formed Deposits (D 993 – 51):[3]

Section 8.—Revise Paragraphs (*h*), (*i*), (*j*), and (*k*) to read as follows, renumbering subsequent notes accordingly:

(*h*) *Iodine, Standard Solution* (*0.01 N*).—Dissolve 0.6346 g of resublimed iodine in a solution of 7.5 g of potassium iodide (KI) and 6 ml of water and dilute to 500 ml in a volumetric flask. Store in dark, glass-stoppered bottle. Standardize against 0.01 *N* $Na_2S_2O_3$ solution (Paragraph (j)).

(*i*) *India Ink.*

(*j*) *Sodium Thiosulfate, Standard Solution* (*0.01 N*).—Dissolve 2.484 g of sodium thiosulfate ($Na_2S_2O_3 \cdot 5H_2O$) in water and dilute to 1 liter in a volumetric flask. Determine the exact normality by titration against a weighed amount of potassium dichromate ($K_2Cr_2O_7$) (Note 1).

NOTE 1.—If frequent restandardization of this solution is to be avoided, it must be stabilized with an inhibitor. One per cent of potassium furoate will limit the decrease in normality to less than 2 per cent in one year, over half of this decrease occurring in the first month. Four tenths per cent of borax has been used for the same purpose.

(*k*) *Starch Indicator.*—Make a paste of 1 g of arrowroot starch or soluble iodometric starch in cold water. Pour the paste into 100 ml of boiling water and boil for several minutes. Store in a glass-stoppered bottle in a cool place. Starch solution prepared in this manner will remain chemically stable for two or three days.

Non-Referee Method B.—Delete this method in its entirety, making the necessary changes in Section 1.

Standard Definitions of Terms Relating to Industrial Water and Industrial Waste Water (D 1129 – 57):[4]

New Definitions.—Insert the following new definitions of terms in their proper place:

Backwash.—That part of the operating cycle of an ion-exchange process wherein a reverse upward flow of water expands the bed, effecting such physical changes as loosening the bed to counteract compacting, stirring up and washing off light insoluble contaminants to clean the bed, or separating a mixed bed into its components to prepare it for regeneration.

Industrial Waste Water.—Water discharged from an industrial process as a result of formation or utilization in that process.

NOTE.—Industrial waste water may have been utilized directly or indirectly, such as cooling water. Industrial waste water may be discharged into other processes, recovery systems, natural streams, or other receiving bodies.

Ion Exchange.—A reversible process by which ions are interchanged between a solid and a liquid with no substantial structural changes of the solid.

Ion-Exchange Capacity.—The weight of ions which can be exchanged by a unit volume of ion-exchange material.

Ion-Exchange Material.—An insoluble solid that has the ability to exchange reversibly certain ions in its structure, or attached to its surface as functional groups, with ions in a surrounding medium.

Minimum Determinability.—The lowest value that can be determined within the stated precision of a method. Minimum determinability is expressed quantitatively in the same dimension that is used for reporting results of a test.

NOTE.—Minimum determinability corresponds to the lower limit of the range of quantitative applicability of a method. It does approach, but cannot equal or be less than, precision of a method. (See Sensitivity.)

Oxidation-Reduction Potential.—The electromotive force developed by a platinum electrode immersed in the water, referred to the standard hydrogen electrode (D 1498).

Regeneration.—That part of the operating cycle of an ion-exchange process in which a specific chemical solution is passed through the ion-exchange bed to prepare it for a service run.

Regeneration Level.—The total weight of regenerant used per unit quantity of ion-exchange material in a single regeneration.

NOTE.—Regeneration level is usually expressed as pounds of regenerant per cubic foot of fully hydrated ion-exchange material in a specific ionic form, measured after backwash and draining.

Sensitivity.—The least amount or concentration that can be detected, not determined, by a method.

NOTE.—A numerical value of sensitivity must be less than either precision or minimum determinability of a given method and in some cases may be only a small fraction of the latter. (See Minimum Determinability.)

Service Run.—That part of the operating cycle of an ion-exchange process in which an industrial or test water is passed through a bed of the ion-exchange material in order to remove specific ions from the water or to exchange them for an equivalent amount of a specific ion from the bed material.

Sodium Cycle.—The operation of a cation exchange cycle wherein the removal of specified ions from the influent water is accomplished by exchange with an equivalent amount of sodium ion from the exchange material.

Surface Tension.—A property arising from molecular forces of the surface film of all liquids which tend to alter the contained volume of liquid into a form of minimum superficial area.

NOTE.—Surface tension is numerically equal to the force acting normal to an imaginary line of unit length in a surface. It is also numerically equal to the work required to enlarge the surface by unit area. The usual unit of measurement is dynes per centimeter.

Water-Borne Industrial Waste.—A water-borne substance (solids, liquids, or gases) resulting from an industrial operation or process and discharged into the water therefrom.

Methods of Test for Fluoride Ion in Industrial Water and Industrial Waste Water (D 1179 – 57):[3] Revise as appended hereto.[2]

Supporting data for this method are given in Appendix I.[5]

REAPPROVAL OF STANDARDS

The committee recommends the reapproval of the following standards which have stood for six years or more without revisions in substance:

Methods of Test for:

- Calcium Ion and Magnesium Ion in Industrial Water (**D 511 – 52**),
- Hydroxide Ion in Industrial Water and Industrial Waste Water (**D 514 – 47**),
- Total Aluminum and Aluminum Ion in Industrial Water (**D 857 – 50**),
- Manganese in Industrial Water and Industrial Waste Water (**D 858 – 49**), and
- Nitrate Ion in Industrial Water (**D 992 – 52**).

Methods of:

- Corrosivity Test of Industrial Water (USBM Embrittlement Detector Method) (**D 807 – 52**),
- Field Sampling of Water-Formed Deposits (**D 887 – 49**),
- Reporting Results of Examination and Analysis of Water-Formed Deposits (**D 933 – 50**),
- Identification of Crystalline Compounds in Water-Formed Deposits by X-Ray Diffraction (**D 934 – 52**), and
- Corrosivity Test of Industrial Water (NDHA Method) (**D 935 – 49**).

Specifications for:

- Substitute Ocean Water (**D 1141 – 52**).

TENTATIVES CONTINUED WITHOUT REVISION

The committee recommends the continuation without revisions in substance of the following tentatives:

Methods of Test for:

- Chloride Ion in Industrial Water and Industrial Waste Water (**D 512 – 55 T**),
- Silica in Industrial Water and Industrial Waste Water (**D 859 – 55 T**),
- Dissolved Oxygen in Industrial Water (**D 888 – 49 T**),
- Iron in Industrial Water and Industrial Waste Water (**D 1068 – 55 T**),
- Sodium and Potassium in Industrial Water (**D 1127 – 50 T**),
- Chloroform-Extractable Matter in Industrial Water and Industrial Waste Water (**D 1178 – 54 T**),
- Sulfides in Industrial Water (**D 1255 – 55 T**),
- Sulfite Ion in Industrial Water (**D 1339 – 54 T**),
- Evaluating Acute Toxicity of Industrial Waste Water to Fresh Water Fishes (**D 1345 – 54 T**), and

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[5] See p. 408.

Sodium and Potassium Ions in Industrial Water and Water-Formed Deposits by Flame Photometry (**D 1428 – 56 T**).

Tentative Scheme for:

Analysis of Industrial Water and Industrial Waste Water (**D 1256 – 53 T**).

EDITORIAL CHANGES

The committee recommends editorial changes as indicated in the following tentatives and standards:

Tentative Methods of Test for Chloride Ion in Industrial Water and Industrial Waste Water (D 512 – 55 T):[3]

Section 9.—In Paragraph (*c*) revise the title to read "*Mercuric Nitrate, Standard Solution* (*0.025 N*)."

Revise the title of Paragraph (*d*) to read "*Mercuric Nitrate, Standard Solution* (*0.0141 N*)." In the first sentence revise "2.5200 g" to read "2.4200 g."

Change Paragraph (*f*) to read as follows:

(*f*) *Nitric Acid* (*1:49*).—Mix 1 volume of concentrated nitric acid (HNO_3, sp gr 1.42) with 49 volumes of water.

Change the title of Paragraph (*h*) to read "*Sodium Chloride, Standard Solution* (*0.025 N*)."

Change Paragraph (*i*) to read as follows:

(*i*) *Sodium Hydroxide Solution* (*10 g per liter*).—Dissolve 10 g of sodium hydroxide (NaOH) in water and dilute to 1 liter.

Footnote 6.—Change to read as follows:

[6] Eastman Kodak No. 4459S diphenylcarbazone and No. 752 tetrabromophenolsulfonephthalein (bromophenol blue) have been found satisfactory for this purpose. This diphenylcarbazone 1 – bromophenol blue indicator, is covered by U. S. Patent No. 2,784,064. By publication of this method the American Society for Testing Materials does not undertake to insure anyone utilizing the method against liability for infringement of Letters Patent nor assume any such liability and such publication should not be construed as a recommendation of any patented or proprietary reagents or procedure that may be involved.

Section 16.—Change Paragraph (*b*) to read as follows:

(*b*) *Phenolphthalein Indicator Solution* (*5 g per liter*).—Dissolve 0.5 g of phenolphthalein in 50 ml of 95 per cent ethyl alcohol. Dilute to 100 ml with water.

Revise the title of Paragraph (*d*) to read "*Silver Nitrate, Standard Solution* (*0.025 N*)."

Change Paragraphs (*e*), (*f*), and (*g*) to read as follows:

(*e*) *Sodium Chloride, Standard Solution* (*0.025 N*).—Dry 1.4613 ± 0.0002 g of sodium chloride (NaCl) for 1 hr at 600 C, dissolve in water, and dilute to 1 liter at 20 C in a volumetric flask (Note 4).

(*f*) *Sodium Hydroxide Solution* (*10 g per liter*).—Dissolve 10 g of sodium hydroxide (NaOH) in water and dilute to 1 liter.

(*g*) *Sulfuric Acid* (*1:19*).—Add 1 volume of concentrated nitric acid (H_2SO_4, sp gr 1.84) to 20 volumes of water and mix.

Standard Method of Test for Nitrate Ion in Industrial Water (D 992 – 52):[3]

Section 7.—Reletter Paragraph (*a*) as Paragraph (*b*) and revise the title to read "*Potassium Nitrate, Standard Solution.*"

Reletter Paragraph (*b*) as Paragraph (*a*).

Tentative Method of Test for Chloroform-Extractable Matter in Industrial Water and Industrial Waste Water (D 1178 – 54 T):[3]

Section 6.—Reletter Paragraph (*a*) as Paragraph (*b*) and change the specific gravity of HCl to read "1.19."

Reletter Paragraph (*b*) as Paragraph (*a*) and delete the word "redistilled."

Section 10.—Change to read as follows, retaining Note 4:

10. Under the accepted definitions of precision and accuracy, values cannot be given because of the nature of the material being isolated (Note 4).

Tentative Method of Test for Sulfite Ion in Industrial Water (D 1339 – 54 T):[3]

Section 6.—Revise the title of Paragraph (*a*) to read "*Starch Indicator Solution.*"

Change Paragraph (*b*) to read as follows:

(*b*) *Hydrochloric Acid* (*1:1*).—Mix 1 volume of concentrated hydrochloric acid (HCl, sp gr 1.19) with 1 volume of water.

Revise the title of Paragraph (*d*) to read "*Potassium Iodate, Standard Solution* (*1 ml = 1 mg* Na_2SO_3)."

Section 8.—Change the first sentence to read "Pour 10 ml of HCl (1:1) into a 250-ml wide-mouth Erlenmeyer flask."

Tentative Methods of Test for Ammonia in Industrial Water and Industrial Waste Water (D 1426–56 T):[6]

Section 9.—Change Paragraph (*c*) to read as follows:

(*c*) *Methyl Red Indicator Solution* (*1 g per liter*).—Dissolve 0.1 g of water-soluble methyl red in water and dilute to 100 ml with water.

Tentative Methods of Test for Residual Chlorine in Industrial Waste Water (D 1427 - 56 T):[6]

Section 4.—In line 7 of Paragraph (*d*), replace the formula given with the words "sodium arsenite."

Delete Paragraph (*h*).

Reletter Paragraph (*i*) as Paragraph (*h*) and change the title to read "*Starch Indicator Solution*."

Tentative Methods of Sampling Homogeneous Industrial Waste Water (D 1496 - 57 T):[4]

Section 3 (*a*).—Revise to read as follows, renumbering subsequent Notes accordingly:

3. (*a*) The terms "industrial waste water" and "water-borne industrial waste" in these methods are defined in accordance with the Definitions of Terms Relating to Industrial Water and Industrial Waste Water (ASTM Designation: D 1129), as follows:

Industrial Waste Water.—Water discharged from an industrial process as a result of formation or utilization in that process (Note 1).

NOTE 1.—Industrial waste water may have been utilized directly or indirectly as cooling water. Industrial waste water may be discharged into other processes, recovery systems, natural streams, or other receiving bodies.

Water-Borne Industrial Waste.—A water-borne substance (solids, liquids, or gases) resulting from an industrial operation or process, and discharged into the water therefrom.

Tentative Method of Test for Iron in High-Purity Industrial Water (D 1497 - 57 T):[4]

Section 10 (*d*).—Continue the last sentence by adding "and make the determination on the test solution diluted to appropriate volume with alcohol."

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[7]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I, Advisory (Max Hecht, chairman) administered the operation of the committee during the year. This included the appointment of symposium chairmen and chairmen of Subcommittees VI and VII; proposals and recommendations made to Headquarters concerning publications sponsored by Committee D-19; and the approval of recommendations submitted by the other subcommittees, explained in more detail in the other subcommittee reports.

Subcommittee II on Definitions and General Specifications (S. K. Love, chairman).—Nearly all of the first drafts of the chapters have been received for the Second Edition of the Manual on Industrial Water. An additional chapter on radioactivity is in preparation. The topical outline of the new chapter was distributed to the committee for comment and letter ballot.

A Task Group on Uniformity of Reagents was organized to reduce the number of reagents used in the methods of Committee D-19. A draft of so-called ground rules is under review.

The scheme for analysis is being revised to reflect the new methods that have been placed on the books. A new format has been adopted.

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[6] 1956 Supplement to Book of ASTM Standards, Part 7.

[7] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

The second draft of a method for calculation of precision is being reviewed.

Definitions were approved for industrial water, minimum determinability, oxidation-reduction potential, sensitivity, surface tension, water-borne industrial waste, and eight terms relating to cation-exchange procedures.

Subcommittee III on Methods of Sampling and Flow Measurement (E. F. Davidson, chairman).—The subcommittee completed the revision of the Tentative Methods of Sampling Homogeneous Industrial Waste Water (D 1496 - 57 T), referred to earlier in the report.

The Task Group on Sampling Industrial Waste Water has drafted a Proposed Method of Sampling Industrial Waste Water Containing Floating Matter.

Final revisions of Chapter VI, Sampling and Flow Measurement of Industrial Water and Waste Water, for the Manual on Industrial Water, were completed.

Subcommittee IV on Methods of Analysis (F. E. Clarke, chairman).—The subcommittee has completed revision of the methods for fluoride ion, hydrazine, and phosphate ion referred to earlier in the report. Numerous existing methods have been revised to improve over-all uniformity of reagent specification and composition in the committee's methods. The subcommittee recommended the adoption as standard of method D 1426 - 56 T. Considerable progress has been made in the development of new methods for high nitrite, micro chloride (colorimetric), lead, nickel, copper, zinc, and sulfate. New projects have been started on methods for aluminum and referee sulfite and on establishment of accuracy in existing dissolved oxygen test methods.

Subcommittee V on Methods of Testing (A. O. Walker, chairman).—The Task Group on Biological Microscope Methods has completed the revisions of Methods D 932, D 993, and D 1128.

The task groups on hydrogen and on surface tension have completed their assignments and proposed tentative methods are appended to the report.[2]

The Task Group on the flame photometer is working on the revisions of the high-purity water procedure to increase the usefulness of the method. It is expected that the sensitivity of the method will be below 1.0 ppb. Revisions are being considered of the method for water-formed deposits.

A procedure for electrical conductivity will be written incorporating the Power Test Code Procedure and added to the present method as a non-referee procedure.

Drafts of proposed methods on color and turbidity have been prepared. These drafts have been submitted to Committee E-12 on Appearance for suggestions and comments.

A method of measurement of gamma activity is being drafted.

Subcommittee VI on Corrosivity and Performance Testing (James K. Rice, chairman).—The Task Group on Ion Exchange has produced a revised draft of a method for the performance testing of cation-exchange materials in the sodium cycle. This method is to be subjected to round-robin testing to determine the precision and to develop any hidden flaws. Work is proceeding concurrently on the checking of a method of test of cation-exchange materials in the hydrogen cycle.

The Task Group on Tubular Metallic Products completed the proposed Tentative Method of Test for the Effect of a Water on Tubular Heat Exchangers, referred to earlier in the report.

The Task Group on Radioactive Tracer Testing has completed a bibliography on radioactive materials in water. The bibliography, started by the late F. N. Alquist, is complete through the end of 1957 and will be maintained up to date with annual supplements. The Task Group plans to prepare an index to the bibliography.

The subcommittee is preparing a proposed Method of Test for the Effect of Mechanical Steam Separation of Steam Impurities. This proposed method is intended for application to steam generation in both conventionally-fired and nuclear-fired steam generators, and for concentrations of less than 0.1 ppm of dissolved solids.

Subcommittee VII on Industrial Waste Water (R. A. Baker, chairman).—A joint task group of subcommittees IV and VII recommended Method D 1293 – 55 T for adoption as standard. Method D 1067 – 57 T was continued as tentative pending reconciliation of definitions.

The Task Group on Chromium has tested modifications of the permanganate procedure necessitated by the use of harbor water containing organic material which caused interference. A silver peroxide method will also be considered which may circumvent this interference.

The Task Group on Cyanides has been continuing round-robin testing of the reflux period and the effect of two catalysts on precision using the ferroferrocyanide complex.

The subcommittee recommended that method D 1069 – 54 T be adopted as standard.

Developmental work is under way to solve problems created by nitrogen compounds and chlorides in Method D 1252 – 55 T. The Methods of Tests for Dissolved Oxygen in Industrial Waste Water[2] were recommended to the society for acceptance as tentative. A method for biochemical oxygen demand is being developed.

The Task Group on Phenols had considered a rapid non-referee infrared method for phenol determination, but since the analytical instruments required are expensive and not available to many laboratories the method was dropped. Low level round-robin testing of the 4-aminoantipyrine method for phenols is in progress.

The Task Group on Specific Gravity had considered the gravitometer measurement of specific gravity as a non-referee procedure but tests proved unfavorable. Method D 1429 – 56 T is currently being tested.

The Task Group on Synthetic Surface Active Materials suspended consideration of the methylene blue and methyl green colorimetric procedures pending completion of a new short method.

Method D 1292 – 53 T is being reviewed and compared with three other procedures.

Method D 1345 – 54 T was recommended for adoption as standard. A procedure for determination of toxicity to diatoms is being developed.

After changes relating to reagents were made, Method D 1427 – 56 T was recommended for adoption as standard.

The choice of solvents in Method D 1340 – 56 is under study. Method D 1178 – 54 T is being compared with procedures developed by other technical groups for possible consolidation.

This report has been submitted to letter ballot of the committee, which consists of 134 members; 73 members have returned their ballots, of whom 70 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

MAX HECHT,
Chairman.

O. M. ELLIOTT,
Secretary.

APPENDIX

SUPPORTING DATA FOR METHODS OF TEST FOR FLUORIDE ION IN INDUSTRIAL WATER AND INDUSTRIAL WASTE WATER

The effect of many ions is given for 0.00 ppm fluoride in Table I and for 1.00 ppm fluoride in Table II.

The following concentrations of foreign ions were added to four selected fluoride standards:

3.3 ppm of Cr^{+6}
5 ppm of Ni, Pb, and Cd
10 ppm of Al, Fe, Mn, Zn, PO_4, and CN
10 000 ppm of Cl

Fluoride Taken, ppm	Fluoride Found, ppm
0.00	0.00
0.50	0.47
1.00	0.98
2.00	1.94

TABLE I.—EFFECT OF IONS FOR 0.00 PPM FLUORIDE (NON-REFEREE METHOD B).

Foreign Ion, ppm	Fluoride Found, ppm	Foreign Ion, ppm	Fluoride found, ppm
100 SiO_2	−0.04	5 Cd	0.02
10 Al	0.02	500 Ca	0.00
10 Fe	−0.03	100 Mg	−0.03
5 Mn	0.00	1 000 HCO_3	−0.04
10 Mn	0.00	2 000 HCO_3	−0.03
0.05 Cr^{+6}	0.00	10 000 Cl	0.00
1.00 Cr^{+6}	0.00	2 000 NO_3	0.02
5.00 Cr^{+6}	−0.03	5 PO_4	0.03
5 Ni	−0.02	10 PO_4	0.14
5 Cu	0.00	10 CN	−0.04
5 Pb	−0.02	10 B_4O_7	0.00
10 Zn	0.03	10 $(OCl)_2$	0.00

Approximately five hundred natural surface and ground water samples of varying types have been analyzed by the method at one U. S. Geological Survey laboratory. Recovery tests applied to randomly selected samples have been satisfactory. The results of ten tests representing widely varied water samples are given in Table III. In each case 1.00 ppm fluoride was added to the water sample. Table IV presents values for precision of the methods.

TABLE II.—EFFECT OF IONS FOR 1.00 PPM FLUORIDE (NON-REFEREE METHOD B).

Foreign Ion, ppm	Fluoride Found, ppm	Foreign Ion, ppm	Fluoride Found, ppm
100 SiO_2	0.97	5 Cd	1.01
10 Al	0.96	500 Ca	1.02
10 Fe	0.97	100 Mg	1.00
5 Mn	0.97	1 000 HCO_3	1.01
10 Mn	0.97	2 000 HCO_3	0.97
0.05 Cr^{+6}	1.00	10 000 Cl	1.00
1.0 Cr^{+6}	0.97	2 000 NO_3	1.01
5.0 Cr^{+6}	1.00	5 PO_4	1.02
5 Ni	0.97	10 PO_4	1.06
5 Cu	1.00	10 CN	0.96
5 Pb	1.00	10 B_4O_7	1.00
10 Zn	1.02	10 $(OCl)_2$	0.97

TABLE III.—RECOVERY TESTS, 1.00 PPM FLUORIDE ADDED (NON-REFEREE METHOD B).

Location	Dissolved Solids, ppm	Significant Constituents, ppm	Initial Fluoride, ppm	Recovered Fluoride, ppm	Recovery, per cent
Woodboro, Md.	42	...	0.15	1.15	100
Rocky Ridge, Md.	306	...	0.05	1.10	105
Antietam, Md	464	HCO_3, 389	0.30	1.27	97
Sumter, S. C.	31	Fe, 1.9	0.17	1.15	98
Chico, Calif.	20 700	SO_4, 3 050	1.80	2.80	100
Colusa, Calif.	15 600	HCO_3, 5 680 SiO_2, 109 Fe, 6.9 NO_3, 254	1.80	2.75	95
Dunsmuir, Calif.	4 420	Fe, 9.4	0.15	1.07	92
Tuscan Spring, Calif.	21 500	H_2S, 172	2.50	3.60	110
Azores Islands	617		0.55	1.55	100
Sistersville, W. Va.	112 000	Fe, 39 Al, 10	1.10	1.95	85

TABLE IV.—PRECISION.

Fluoride, ppm	Standard Deviation	
	Precision, ppm	
	Non-Referee Method A[a, b]	Non-Referee Method B[a]
0.00	0.01	0.00
0.50	0.01	...
1.00	0.02	0.01
2.00	...	0.02
2.50	...	0.01
Average	0.01	0.01

[a] Based on the undiluted samples of 100 ml for Non-Referee Method A and 10 ml for Non-Referee Method B.

[b] Additional supporting data pertinent to the zirconium-alizarin (Non-Referee Method A) are given in Appendix I to the 1955 Report of Committee D-19, *Proceedings*, Am. Soc. Testing Mats., Vol. 55, p. 548 (1955).

REPORT OF COMMITTEE D-20

ON

PLASTICS*

Committee D-20 on Plastics held three meetings during the year: at Atlantic City, N. J., on June 17 to 19, 1957; at Virginia Beach, Va., on November 20 to 22, 1957; and at Pittsburgh, Pa., on February 24 to 26, 1958.

The committee consists of 559 members, of whom 218 are classified as producers, 142 as consumers, and 199 as general interest members. There are 212 committee voting members.

The officers elected for the ensuing term of two years are as follows:

Chairman, F. W. Reinhart.
First Vice-Chairman, A. C. Webber.
Second Vice-Chairman, J. B. DeCoste.
General Secretary, R. M. Berg.
Membership Secretary, R. H. Carey.

The following appointments were made:

Meeting Secretary, B. L. Lewis.
Financial Secretary, F. Y. Speight.

A new Subcommittee XIII on Statistical Techniques was established. It is planned to investigate the need for the establishment of a new Subcommittee on Plastics Tooling (tools made of plastics). A procedure for reviewing test methods and specifications being prepared or issued by governmental agencies and other societies was developed and is being used on a trial basis. A revision of the by-laws is under way. The orientation course to indoctrinate new officers and members has been successful and will be continued.

International Standardization Activities.—The United States participates in the work of Technical Committee ISO/TC 61 on Plastics of the International Organization for Standardization (ISO), through its American Group, C. H. Adams, Leader. Representatives from the United States attended the seventh meeting of ISO/TC 61 on Plastics at Bürgenstock, Switzerland, on July 8 to 13, 1957. Thirty-nine test procedures and a group of definitions were dealt with during the year. Proposed methods for impact strength, thermal stability of poly(vinyl chloride) compounds, bleeding of colorants, and incandescence resistance were completed.

Action was taken on 16 methods of test, bringing to 24 the total of Draft ISO Recommendations that have been developed by the Committee. The most significant action of the year was the development by ATCO (coordinating committee on atmospheric conditioning for testing) of three testing and conditioning atmospheres following, in essence, the recommendations of ISO/TC 61 made at the Hague in 1956, as follows: 20 C and 65 per cent RH, 27 C and 65 per cent RH, and 23 C and 50 per cent RH (ASTM Methods D 618). Another highlight was the completion of the monumental task of compiling a list of equivalent terms in French, English, and Russian pertaining to plastics technology. Work in progress includes: methods of test for dynamic properties, creep, acetone-soluble matter in phenolic molding materials, viscosity of polyamide resins, resistance of plastics to fungus attack and standard die design for producing test specimen bars. A detailed report on the year's activity may be found in the *Magazine of Standards*, ASA, January 1958. A report of the meeting in Switzerland appeared in the October 1957 issue of the ASTM BULLETIN.

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* Sixty-first Annual Meeting of the Society, June 22–27, 1958.

The 1958 meeting of ISO 61 on Plastics will be held in Washington November 3 to 8. Delegates have been invited to participate in a symposium on plastics testing and standardization sponsored by Committee D-20 preceding the ISO 61 meeting.

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1957 Annual Meeting, Committee D-20 presented to the Society through the Administrative Committee on Standards the recommendations listed below. On the dates indicated the recommendations were accepted by the administrative committee. The new and revised tentatives will appear in the 1958 Book of ASTM Standards, Part 9.

New Tentative Method of Test for:

Diffuse Light Transmission Factor of Reinforced Plastic Panels (D 1494 - 57 T) (Approved Dec. 13, 1957),

Vicat Softening Point (D 1525 - 58 T) (Approved June 18, 1958),

Bierbaum Scratch Hardness of Plastic Materials (D 1526 - 58 T) (Approved June 18, 1958), and

Tensile Modulus of Elasticity of Thin Plastic Sheeting (D 1530 - 58 T) (Approved June 18, 1958).

New Tentative Specifications for:

Biaxially Oriented Styrene Plastic Sheet (D 1463 - 58 T) (Approved Feb. 28, 1958).

New Tentative Specifications and Methods of Test for:

Random Chopped Glass Fiber Reinforcing Mat (D 1529 - 58 T) (Approved June 18, 1958).

Revision of Tentative Specifications for:

Polyester Compounds (D 1201 - 52 T) (Approved Feb. 28, 1958).

Revision of Tentative Method of Test for:

Flexural Properties of Plastics (D 790 - 49 T) (Approved June 18, 1958).

Revision and Reversion to Tentative of Standard:

Specifications for Urea Formaldehyde Molding Compounds (D 705 - 55) (Approved Feb. 28, 1958),

Method of Test for Dilute Solution Viscosity of Vinyl Chloride Polymers (D 1243 - 54) (Approved June 18, 1958), and

Method of Test for Stiffness in Flexure of Plastics (D 747 - 50) (Approved June 18, 1958).

RECOMMENDATIONS AFFECTING STANDARDS

The committee is presenting for publication eleven new tentatives and is recommending the revision of six tentatives, the adoption as standard of five tentatives, and the reaffirmation of ten standards. One proposed method is being recommended for publication as information. The new tentatives, the standards affected, together with the revisions recommended are given in detail in the Appendix.[1]

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[2]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Mechanical Properties (C. H. Adams, chairman) prepared a revision of the Tentative Method of Test for Tensile Properties of Plastics (D 638 - 56 T) incorporating changes in testing speeds. Work is in progress on a method for microtensile testing and on revisions to the definitions appended to Method D 638. Basic research on a tensile impact method is continuing as a result of an earlier round robin. Film impact is concerned with projectile and free fall techniques. A revision of the Tentative Method of Test for Flexural Properties of Plastics (D 790 - 49 T) has been sent to the Administrative Committee on Standards. The Tentative Method of Test for Determination of Bierbaum Scratch Hardness of Plastic Materials was prepared, and also the new Tentative Method of Test for Tensile Modulus of Elasticity of Thin Plastic Sheeting. A new method for the blocking resistance of films is under study. The subcommittee revised the Method of Test for Stiffness in Flexure of Plastics (D 747 - 50). Joint jurisdiction with Committee D-11 on Methods D 676 and D 1484 has yet to be established. Recommendations that would make the methods more pertinent to plastics have been sent to Committee D-11.

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[1] See p. 417.

[2] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

Subcommittee II on Effects of Radiation (D. S. Ballantine, chairman), a joint subcommittee with Committee D-9, was reorganized and now contains five sections: Section A on Nomenclature and Definitions, Section B on Dosimetry, Section C on Radiation Exposure Methods, Section D on Education, and Section E on Research.

A glossary of nuclear terms and definitions, based on the much larger ASME glossary, is being prepared for possible inclusion in the D-9 and D-20 Compilations of Standards. A dosimetry method using the widely accepted ferrous sulfate chemical dosimeter is being completed and will be proposed for a round-robin testing program. A recommended practice for exposure of materials to radiation is being written by Section C. A series of lectures has been started covering diverse aspects of radiation effects. These will be given at regular meetings of Committees D-9 and D-20, and may be published in the future. The Section on Research has invited suggestions of subjects of broad general interest to members of ASTM for a future integrated research program.

Subcommittee III on Thermal Properties (R. M. Berg, chairman).—A change in title for Method D 648 has been developed in the subcommittee and recommended to committee D-20 for adoption. The new title is based on the ISO title for virtually the same method, and its adoption should facilitate international standardization. The ZST (zero-strength-time) test has been shown to be reliable and reproducible when applied to polyethylene, and further work is in progress to determine the correlation of ZST with intrinsic viscosity and to determine the effect of temperature on ZST. The flammability section has made excellent progress in developing a proposed tentative method of test for measuring the surface flammability of plastics using a radiant heat energy source. Round-robin testing of methods for the flammability of plastic foams and for the ignition temperature of plastics are in process. Test methods for determining the flow characteristics of polyethylene utilizing a high shear capillary viscometer are being studied. Further study of the SPI test for low-temperature brittleness of plastic films continues. A method of test for the Vicat softening point was developed and accepted by Committee D-20. A new section was formed to standardize a method of test for tensile heat distortion temperature of plastic sheeting.

Subcommittee IV on Optical Properties (W. E. Brown, chairman) has collected visual and instrumental data on clarity (see-through) of films, yellowness, and hiding power. Visual methods have generally confirmed the instrumental methods under consideration but will not be studied further because of the operator variables that appear in subjective procedures. Although methods for each of the above properties have been shown to be practical, additional interlaboratory work will be necessary to improve reproducibility sufficiently to permit writing of tentative methods of test.

Contact has been established with the Illuminating Engineering Society Committee on Testing Procedures, particularly for participation in work on hiding power. Liaison representation may result.

The Method of Test for Light Transmission of Corrugated Plastic Panels developed by Subcommittee XVIII and the Society of the Plastics Industry has been reviewed. Agreement was reached on nomenclature and on the method of test such that the method is acceptable in light of basic optical measurements.

Studies are planned to show the applicability of Methods of Test for 60-deg 2-Parameter Gloss. The color difference methods developed by Committee D-1 on Paints will be included in the next edition of the Compilation of ASTM Standards on Plastics. No further work is needed to adapt these methods to plastics.

Subcommittee V on Permanence Properties (B. G. Achammer, chairman) is conducting a round-robin test on an acrylic plastic base containing a yellow dye for use as a fading standard to determine equivalent amounts of exposure in artificial weathering machines. A proposed Tentative Recommended Practice for Operating Light- and Water-Exposure Apparatus (Carbon-Arc Type) for Exposure of Plastics and Elastomeric Materials was written and is being studied. The Tentative Recommended Practice for Resistance of Plastics to Artificial Weathering Using Fluorescent Sun Lamp and a Fog Chamber (D 1501 – 57 T) was published.

Revisions of the Tentative Method of Test for Resistance to Chemical Reagents (D 543 – 56 T); Tentative Method of Test for Water Absorption of Plastics (D 570 – 54 T); and Method of Test for Volatile Loss of Plasticizers from Plastic Materials (D 1203 – 55), were initiated.

Two new sections were formed. Section K on Vinyl Staining will develop a method of test to determine staining of poly(vinyl chloride). One purpose of this method will be to determine staining of vinyl floor tile. Section M on Effect of Microorganisms will develop a method for evaluating the resistance of plastics to microorganisms.

Subcommittee VII on General Analytical Methods (M. R. Rector, chairman) prepared a method for the determination of carbon black content of polyethylene which is to be submitted to ballot. In the area of general physical properties, revisions are in progress on the Tentative Method of Test for Measurement of Density of Plastics by the Density Gradient Technique (D 1505) and on Methods of Test for Specific Gravity of Plastics (D 792). A revision of Method D 1243 for the dilute solution viscosity of poly(vinyl chloride) was completed. A number of other tests on poly(vinyl chloride) resin are being developed for incorporation in a specification being written on this resin by Subcommittee XV. These include test methods on dry blending and plasticizer absorption, paste resin viscosity, bulk density, electrical properties, fish-eyes, and particle size.

Subcommittee VIII on Research (A. G. H. Dietz, chairman) has continued to arrange for presentation of papers on subjects of current interest to the committee.

At the fall meeting four papers were presented:

"Properties of Flake Glass as Reinforcement for Plastics" by John Gibbud of the Owens-Corning Fiberglas Corp.

"A Survey of Statistical Methodology Applicable to Environmental Stress Cracking Data Analysis" by W. M. Chow and D. E. Gould of the Bakelite Co.

"A New Rheometer for Thermoplastics" by

E. H. Merz and R. E. Colwell of the Plastics Division, Monsanto Chemical Co.

"Behavior of Polymethyl Methacrylate Plastics Under Uniform Load" by Professor F. J. McGarry of the Massachusetts Institute of Technology.

At the spring meeting the program was as follows:

"Test Method for Determining Environmental Stress Cracking Resistance of Films" by L. R. Petrone and D. E. Gould of the Bakelite Co.

"The Use of the Torsion Pendulum to Measure Crystallinity and Void Content of Objects made from TFE-Fluorocarbon Resin" by N. G. McCrum of the Polychemicals Dept., E. I. duPont de Nemours & Co., Inc.

"Viscosity-Molecular Weight Relationships" by W. E. Gibbs of the Goodyear Tire and Rubber Co.

Subcommittee IX on Molds and Molding (G. H. Williams, Jr., chairman) has a round robin in progress for the development of an injection molding procedure for type 3 polystyrene in Specification D 703. Small injection molding machines are being studied for the preparation of plastic test specimens. Sections B and D are coordinating their investigations on recommended practices for molding phenolic test specimens.

A new Section on Mold Release is being organized and a program for the development of a method of test for measuring mold release properties is planned.

Subcommittee X on Definitions, Nomenclature and Significance of Tests (By R. J. Bourke for P. E. Willard, chairman) is continuing to add to and to revise the Tentative Definitions of Terms Pertaining to Plastics (D 883 – 57 T) and Tentative Nomenclature of Descriptive Terms Pertaining to Plastics (D 675 – 57 T) so as to keep definitions and nomenclature current.

Definitions for 20 new terms were approved during the year. Revised definitions for "Optical Distortion" and "Pinhole" were approved for inclusion in Nomenclature D 675. The new Tentative Abbreviations of Terms Relating to Plastics is being recommended for publication as tentative. The above items are appended to this report.

Definitions for 19 new terms and revisions for 3 existing terms are in various stages of consideration.

Subcommittee XI on Editorial Review (G. M. Kline, chairman) is reviewing proposed standards and revisions on the subcommittee level. This procedure is expected to result in an over-all improvement in the standards.

Subcommittee XIV on Conditioning (G. M. Armstrong, chairman), a joint committee with Committee D-9 on Electrical Insulating Materials, is working on a new revision of Standard Methods of Conditioning Plastics and Electrical Insulating Materials for Testing (D 618 – 54) that will incorporate a system of nomenclature similar to that used in MIL-P specifications for conditioning procedures and include changes in conditioning atmospheres which will bring the standard into essential agreement with the recommendations of ATCO (ISO coordinating committee on atmospheric conditioning for testing). These recommendations have been accepted by ISO/TC 61 and IEC/TC-15; they have also appeared in recent drafts of British and German conditioning standards.

Subcommittee XV on Thermoplastic Materials (A. C. Webber, chairman) has sent three new material specifications and two revisions to Committee D-20 letter ballot: Cellulose Propionate Molding and Extrusion Materials; Polymethylstyrene Molding and Extrusion Materials; Methylstyrene - Acrylonitrile Copolymer Molding and Extrusion Materials, revision of Specifications and Methods of Test for Polychlorotrifluoroethylene Molding and Extrusion Materials (D 1430); and the very important revision of Specifications for Polyethylene Molding and Extrusion Materials (D 1248 – 52 T).

An extensive revision of the Tentative Specifications for Nylon Injection Molding and Extrusion Compositions (D 789) and a new specification for PTFE Rod are completed and ready for subcommittee ballot. Section work is nearly completed on a new specification for ABS plastics and styrene-butadiene molding and extrusion materials. Section H on Vinyl Resins has joined forces with Section E of Subcommittee VII to develop methods of test needed for writing the specification.

Task groups of Section J on Polyethylene have been very active, developing methods for characterization of polyethylene such as the method for Dilute Solution Viscosity. After four extensive round robins, it has been concluded that the proposed stress-crack method is suitable only for routine inspection and has been published as information only. Work on annealing and tests for oxidative stability is continuing.

Subcommittee XVI on Thermosetting Materials (G. D. Johnston, chairman) has completed a revision of the electrical properties of Specifications D 705 – 55. A proposed allyl material specification is still under consideration. A revision of Specifications D 1201 by the addition of a type 6 is under consideration. Section E on Casting Materials is starting work on epoxy materials.

Subcommittee XVII on Plastic Pipe and Fittings (George H. Reed, chairman) is a joint activity of the SPI and ASTM Committee D-20. The subcommittee consists of approximately 120 members, who comprise six sections working on dimensional and performance requirements and test methods for acrylonitrile-butadiene-styrene (ABS), polyethylene, rigid poly(vinyl chloride), and cellulose acetate butyrate pipe and fittings.

The subcommittee has presently established Tentative ASTM Standards on ABS Plastic Pipe (IPS Dimensions); ABS Pipe (SWP Dimensions), and Cellulose Acetate Butyrate Pipe (SWP Dimensions).

A proposed Tentative Method of Time-to-Failure of Plastic Pipe under Long-Term Hydrostatic Pressure and a Proposed Tentative Method of Test on Time-to-Failure of Plastic Pipe under Short-Term Hydrostatic Pressure are presently being circulated by letter ballot.

A proposed Standard on Flexible Polyethylene Pipe will shortly be sent to letter ballot.

Agreement has now been reached on dimensions for Rigid PVC Pipe and a letter ballot on this proposed standard will shortly be sent to the membership.

The development of proposed dimensional standards on each type of plastic fitting is now under way within Subcommittee XVII. Test methods for creep rupture are also being developed at this time.

Subcommittee XVIII on Reinforced Plastics (Ray B. Crepps, chairman) is working on a specification for reinforced plastic pipe fittings. A tentative specification for woven glass fabric, cleaned and after-finished, for plastic laminates is nearing the final stages for establishment as an ASTM method. A Tentative Specification and Method of Test for Random Chopped Glass Fiber Reinforcing Mat has been approved as an ASTM Tentative. Progress is being made on the development of methods of tests for (*a*) consistency of liquid epoxy and polyester resins, (*b*) epoxy content, and (*c*) curing characteristics of liquid polyesters.

Subcommittee XIX on Plastic Film and Sheeting (T. D. Mecca, retiring chairman) has submitted a specification on non-rigid vinyl chloride plastic sheeting for letter ballot. Earlier work on a rigid vinyl chloride sheeting specification is being reviewed with the intention of preparing a draft for subcommittee ballot.

Test methods for impact strength and flatness of polyethylene film were submitted to Committee D-20 letter ballot. It was decided to discontinue the preparation of a proposed test method of heat sealability.

A proposed specification is being prepared on extruded acrylic sheet for which requirements on internal stress and strain are being considered. A revision of Specifications D 702 on cast methacrylate sheet was prepared.

A specification on biaxially oriented polystyrene sheet was accepted by the Administrative Committee on Standards and a new specification for styrene-butadiene sheet is being prepared.

Subcommittee XX on Cellular Plastics (I. J. Stoneback, chairman) during the past year has condensed its activities into three sections dealing with test methods, polystyrene, and nomenclature. Standards for chemical analysis of raw materials for foams, test methods for moisture vapor transmission, thermal conductivity, flammability, and coefficient of thermal expansion are actively under consideration in Section A on Test Methods. This section also has balloted on methods of testing compression, tension, density, and shear. A task force in Section E on Polystyrene prepared and distributed for comment a specification on expanded and unexpanded beads and polystyrene foams. The members are balloting whether to split the specification three ways to cover expanded beads, foam made from beads, and commercially expanded foam. Section F on Nomenclature has a number of terms under consideration.

This report has been submitted to letter ballot of the committee, which consists of 212 voting members; 123 members returned their ballots, of whom 106 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

F. W. REINHART,
Chairman.

J. B. DECOSTE,
Secretary.

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee D-20 presented to the Society through the Administrative Committee on Standards the following recommendations:

New Tentative Specifications for:

Dimensions of Iron Pipe Size (IPS) Extruded Acrylonitrile-Butadiene-Styrene (ABS) Plastic Pipe (D 1527 – 58 T),
Dimensions of Solvent Welded (SWP Size) Extruded Acrylonitrile-Butadiene-Styrene (ABS) Plastic Pipe (D 1528 – 58 T), and
Cellulose Propionate Molding and Extrusion Compounds (D 1562 – 58 T).

These recommendations were accepted by the Standards Committee on September 9, 1958. Specifications D 1527 and D 1528 appear in the 1958 Book of ASTM Standards, Part 9; Specification D 1562 is available as a separate reprint.

APPENDIX

RECOMMENDATIONS AFFECTING STANDARDS ON PLASTICS

In this Appendix are listed thirteen new tentatives and seven proposed revisions in certain tentatives which are referred to earlier in this report. These standards and tentatives which are being revised appear in their present form in the 1955 Book of ASTM Standards, Part 6, or in the 1956 and 1957 Supplements to Book of ASTM Standards, Part 6.

PROPOSED METHOD TO BE PUBLISHED AS INFORMATION

The committee recommends for publication as information only the Proposed Method of Test for Measuring the Surface Flammability of Plastics Using a Radiant Heat Energy Source, as appended hereto.[1]

NEW TENTATIVES

The committee recommends for publication as tentative the following specifications and methods of test as appended hereto:[2]

Abbreviations of Terms Relating to Plastics:

The designation of plastics and raw materials used in plastics by abbreviations has become common practice and there is a need for standardization.

Specifications for Glass Fabric Reinforced Epoxy Resin Laminates

Method of Test for Dilute Solution Viscosity of Ethylene Polymers:

Solution viscosity is recognized as an important research and development tool for ethylene polymers. It can furnish useful information on a series of homologous polymers.

Method of Test for Bearing Load of Corrugated Plastics Panels:

This specification is the outgrowth of work started by the Society of The Plastics Industry. The objective has been to afford information concerning panels when fasteners are used in installation.

Method of Test for Carbon Black in Ethylene Plastics:

This method is needed for establishing specification requirements on weather-resistant grades of black polyethylene.

Method of Test for Measurement of Flatness of Plastics Sheet or Tubing

Method of Test for Time-to-Failure of Plastic Pipe Under Long-Term Hydrostatic Pressure:

This method is needed for establishing specification requirements on plastic pipe. The method has been rewritten to take into account criticism received on a previous Committee D-20 ballot.

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[1] The proposed method of test appears in the 1958 Compilation of ASTM Standards on Plastics.

[2] The new and revised tentatives appear in the 1958 Book of ASTM Standards, Part 9.

Method of Test for Short-Time Rupture Strength of Thermoplastic Pipe, Tubing, and Fittings

Specifications for Nonrigid Vinyl Chloride Plastic Sheeting

Specification for Methylstyrene-Acrylonitrile Copolymer Molding and Extrusion Material

Specification for Polymethylstyrene Molding and Extrusion Material

Revisions of Tentatives

The committee recommends revisions as indicated of the following tentatives and their continuation as tentative:

Tentative Method of Test for Tensile Properties of Plastics (D 638 - 56 T):

This revision grew out of a need to modify the speed of testing. Other changes involving conditioning and modulus determination have also been made.

Section 1.—Change the words "test specimens of standard shape" to read "standard test specimens."

Section 3.—Delete the first paragraph.

Section 4.—Change Paragraph (*b*) to read as follows:

(*b*) *Extension Indicator.*—A suitable instrument for determining the distance between two fixed points located within the gage length of the test specimen at any time during the test. It is desirable, but not essential, that this instrument automatically record this distance (or any change in it) as a function of the load on the test specimen or of the elapsed time from the start of the test, or both. If only the latter is obtained, load-time data must also be taken. This instrument shall be essentially free of inertia lag at the specified speed of testing and shall be accurate to ±1 per cent of strain or better (Note 9).

Delete Paragraph (*d*).

Section 5(*e*).—Change to read as follows:

(*e*) If it is necessary to place gage marks on the specimen, this shall be done with a wax crayon or India ink that will not affect the material being tested. Gage marks shall not be scratched, punched, or impressed on the specimen.

Section 6.—Delete and replace with the following:

6. *"Standard" Conditioning Procedure.*—The test specimens shall be conditioned and tested in a room or enclosed space maintained at 23 ± 1 C (73.4 ± 1.8 F) and 50 ± 2 per cent relative humidity in accordance with Procedure A of ASTM Method D 618, unless otherwise specified.

Section 7.—Delete Paragraph (*d*).

Section 8.—Change Paragraph (*b*) to read as follows:

(*b*) The speed of testing shall be chosen from one of the following:

Speed A	0.05 in. per min
Speed B	0.20 to 0.25 in. per min
Speed C	2.0 in. per min
Speed D	20 in. per min

The speed of testing shall be determined by the specification for the material being tested, or by agreement between those concerned. When the speed of testing is not specified, a speed of 0.20 to 0.25 in. per min shall be used.

Change Paragraph (*c*) to read as follows:

(*c*) If modulus values are being determined, the speed of testing shall be 0.20 to 0.25 in. per min except for molded or laminated thermosetting materials which shall be tested at a speed of 0.05 in. per min (Note 10). Modulus determinations shall be obtained using separate specimens from those used to determine tensile strength and elongation whenever the speeds of testing are not the same.

Section 9.—Delete Paragraph (*a*), renumbering the subsequent paragraphs accordingly.

Change Paragraph (*d*) to read as follows:

(*d*) If modulus values are being determined, the procedure shall be as follows:

(*1*) Attach the extension indicator.

(*2*) Set the speed of testing at either 0.05 or 0.20 to 0.25 in. per min (see Section 8(*c*)) and start the machine.

(*3*) Record loads and corresponding deformations at appropriate even intervals of strain.

(*4*) Record the load carried by the specimen when the strain reaches 0.02 and the elapsed time from the start of the test until this point is reached. If rupture occurs before the strain reaches 0.02, record the elapsed time from the start of the test until the specimen breaks.

In Paragraph (*d*), delete Items (*5*), (*6*), and (*7*).

Change Paragraph (*e*) and Item (*1*) to read as follows:

(*e*) Tensile strength and elongation shall be determined by the following procedure:

(*1*) Set the speed of testing at either 0.05, 0.20 to 0.25, 2.0, or 20 in. per min (see Section 8(*b*)) and start the machine.

Section 10.—Change Paragraph (*e*) to read as follows:

(*e*) *Elastic Modulus* shall be obtained by extending the initial linear portion of the load-extension curve and dividing the difference in stress corresponding to a certain section on this straight line by the corresponding difference in strain. The result shall be expressed in pounds per square inch and reported to three significant figures.

Explanatory Note 9.—Delete the present Note 9 and substitute "NOTE 9.—Reference is made to Method of Verification and Classification of Extensometers (ASTM Designation: E 83)."

Explanatory Notes 10, 11, 12, and 13.—Delete these Notes and replace with a new Note 10 to read as follows:

NOTE 10.—The lower speed of testing is used for molded or laminated thermosetting material to insure that the response of the extension indicated is sufficient for these high modulus materials.

Tentative Nomenclature of Descriptive Terms Pertaining to Plastics (D 675 - 57 T): Revise the following definitions to read as shown:

Optical Distortion.—Any apparent alteration of the geometric pattern of an object when seen either through a plastic or as a reflection from a plastic surface.

Pinhole.—Very small hole through a plastic.

Tentative Specifications for Cast Methacrylate Plastic Sheet, Rods, Tubes, and Shapes (D 702 - 56 T):

Table II.—Change the displacement factor (optical), max, to read as follows:

	Type I, General Purpose	Type II, General Purpose
Displacement factor (optical), max:[a]		
0.060 to 0.500 in. thickness	50	50
0.625 to 1.000 in. thickness	80	80
1.125 to 2.000 in. thickness	125	125

This change is proposed because the material available commercially does not meet the present requirement and exception is taken to this requirement when supplying sheet under this specification.

Tentative Definitions of Terms Relating to Plastics (D 883 - 57 T):

Add the following new definitions:

Vacuum Forming.—A forming process in which a heated plastic sheet is changed to a desired shape by causing it to flow by reducing the air pressure on one side of the sheet.

Sprue, *n.*—(*1*) The primary feed channel that runs from the outer face of an injection or transfer mold, to the mold gate in a single cavity mold or to the runners in a multiple cavity mold.

(*2*) The piece formed in a primary feed channel or sprue. (See first definition.)

Runner, *n.*—(*1*) The secondary feed channel that runs from the inner end of the sprue in an injection or transfer mold, to the cavity gate.

(*2*) The piece formed in a secondary feed channel or runner. (See first definition.)

Telomer, *n.*—An addition polymer in which the growth of the chainlike molecules is intentionally terminated by radicals supplied by a chain transfer agent.

Specimen, *n.*—An individual piece or portion of a sample used to make a specific test. Specific tests usually require specimens of specific shape and dimensions.

Finishing, *n.*—The removal of defects or the development of desired surface characteristics on plastics products.

Melamine Plastics.—Plastics based on resins made by the condensation of melamine and aldehydes.

Urea Plastics.—Plastics based on resins made by the condensation of urea and aldehydes.

Epoxy Plastics.—Plastics based on resins made by the reaction of epoxides or oxiranes with other materials such as amines, alcohols, phenols, carboxylic acids, acid anhydrides, and unsaturated compounds.

Autothermal Extrusion.—A method of extrusion in which the conversion of the drive energy, through friction, is the sole source of heat.

Oligomer, *n.*—A polymer consisting of only a few monomer units such as a dimer, trimer, tetramer, etc., or their mixtures.

Fabricating, *n.*—The manufacture of plastic products from molded parts, rods, tubes, sheeting, extrusions, or other forms by appropriate operations such as punching, cutting, drilling, and tapping. Fabrication includes fastening plastic parts together or to other parts by mechanical devices, adhesives, heat sealing, or other means.

Weathering, *n.*—The exposure of plastics outdoors.

Sample, *n.*—A small part or portion of a plastic material or product intended to be representative of the whole.

Glass Finish.—A material applied to the surface of glass fibers used to reinforce plastics and intended to improve the physical properties of such reinforced plastics over that obtained using glass reinforcement without finish.

Polymethylenic, *adj.*—Pertaining to a type of molecular structure consisting of a series of methylene groups.

Viscosity, *n.*—The property of resistance to flow exhibited within the body of a material.

NOTE.—This property can be expressed in terms of the relationship between applied shearing stress and resulting rate of strain in shear. Viscosity is usually taken to mean "Newtonian viscosity," in which case the ratio of shearing stress to the rate of shearing strain is constant. In non-Newtonian behavior, which is the usual case with plastic materials, the ratio varies with the shearing stress. Such ratios are often called the "apparent viscosities" at the corresponding shearing stresses. (See Viscosity Coefficient.)

Viscosity Coefficient.—The shearing stress necessary to induce a unit velocity flow gradient in a material.

NOTE.—In actual measurement, the viscosity coefficient of a material is obtained from the ratio of shearing stress to shearing rate. This assumes the ratio to be constant and independent of the shearing stress, a condition which is satisfied only by Newtonian fluids. Consequently, in all other cases, values obtained are apparent and represent one point on the flow curve. In the metric system, the viscosity coefficient is expressed in poises, units being dyne sec per sq cm. (See Viscosity.)

Tentative Specifications for Polyethylene Molding and Extrusion Materials (D 1248 – 52 T):

The proposed revision, appended hereto,[2] takes into account new classes of polyethylene that have appeared on the market. Density has been chosen as the primary parameter for classification.

Tentative Specifications and Methods of Test for Polychlorotrifluoroethylene Molding and Extrusion Materials (D 1430 – 56 T):

Table I.—Delete the line "Apparent molecular weight.[a]" Revise footnote *a* and add new footnotes *b* and *c* to read as indicated. The column headed "Grade 4" shall be replaced with two columns as follows:

	Grade 4	Grade 5
Specific gravity, 23/23 C (73.4/73.4 F), min.	2.10	2.10
Zero strength time, sec[a]	751–2500	>2500
Solution viscosity, centistokes[a]	[b]	[b]
Flow index, mg per min[a]	[c]	[c]
Deformation under load, max (at 250 lb, 24 hr, 70 ± 1 C), per cent	10	10
Dielectric constant, max:		
10^3 cps	2.70	2.70
10^6 cps	2.50	2.50
Dissipation factor, max:		
10^3	0.030	0.030
10^6	0.012	0.012

[a] The material shall be characterized by any one of the zero strength time, solution viscosity, and flow index methods, as specified by the purchaser in the contract or order.

[b] Solution viscosity measurements in this range are extremely difficult.

[c] Flow index is insensitive in this range.

Section 7.—Change the heading of Paragraph (*d*) to read "*Zero Strength Time.*" Change the Note in this paragraph to read as follows:

NOTE.—Close control of molecular weight of these polymers is necessary because the fabricating temperatures are very high and close to the point of rapid thermal degradation. The test for zero strength time, as described in Paragraph (*3*), is well suited to this type of control, as it is rapid, simple, and adaptable to semi-automatic operation.

Change the heading of Paragraph (*e*) to read "*Solution Viscosity.*"

Change the heading of Paragraph (*f*) to read "*Flow Index.*"

ADOPTION OF TENTATIVES AS STANDARD WITHOUT REVISION

The committee recommends that the following tentatives be approved for reference to letter ballot of the Society for adoption as standard without revision:

Tentative Method of Test for:

Flammability of Flexible Thin Plastic Sheeting (**D 1433 - 56 T**), and
Gas Transmission Rate of Plastic Sheeting (**D 1434 - 56 T**).

Tentative Recommended Practice for:

Outdoor Weathering of Plastics (**D 1435 - 56 T**).

Tentative Method of:

Sampling and Testing Plasticizers Used in Plastics (**D 1045 - 55 T**).

Tentative Specifications for:

Nonrigid Poly(Vinyl Chloride) and Copolymer Molding Materials (**D 1432 - 56 T**).

REAPPROVAL OF STANDARDS

The committee recommends the reapproval of the following standards which have stood for six or more years without revision:

Standard Methods of Test for:

Index of Refraction of Transparent Organic Plastics (**D 542 - 50**),
Haze and Luminous Transmittance of Transparent Plastics (**D 1003 - 52**),
Surface Irregularities of Flat Transparent Plastic Sheets (**D 637 - 50**),
Deviation of Line of Sight Through Transparent Plastics (**D 881 - 48**).

Standard Methods of:

Measuring Shrinkage from Mold Dimensions of Molded Plastics (**D 955 - 51**), and
Measuring Changes in Linear Dimensions of Plastics (**D 1042 - 51**).

Recommended Practice for:

Molding Specimens of Amino Plastics (**D 956 - 51**),
Determining Permanent Effect of Heat on Plastics (**D 794 - 49**),
Determining Mold Surface Temperature of Commercial Molds for Plastics (**D 957 - 50**), and
Determining Temperature of Standard ASTM Mold for Test Specimens of Plastics (**D 958 - 57**).

EDITORIAL CHANGES

The committee recommends editorial changes as follows in the Tentative Method of Test for Heat Distortion Temperature of Plastics (**D 648-56**):

Title.—Change the title of the method to read: "Tentative Method of Test for Deflection Temperature of Plastics Under Load."

It is generally agreed that the present title is misleading. The new ISO title for virtually the same method is much clearer and in the interest of international standardization and simplification, Subcommittee III is recommending the new title based on the ISO title be accepted.

REPORT OF COMMITTEE D-21

ON

WAX POLISHES AND RELATED MATERIALS*

Committee D-21 on Wax Polishes and Related Materials held one meeting during the year: at New York City on January 21, 1958. In addition, the subcommittees met in Cincinnati, Ohio, on May 22, 1958.

Additions and changes during the year brought the total membership of the committee to 71, of whom 35 are classified as producers, 13 as consumers, and 23 as general interest members, with one consulting member.

The officers elected for the ensuing term of two years are as follows:

Chairman, W. W. Walton.
Vice-Chairman, J. V. Steinle.
Secretary, B. S. Johnson.

TENTATIVES CONTINUED WITHOUT REVISION

The committee recommends the continuation without revision of the following tentatives:

Tentative Methods of Test for:

Nonvolatile Matter (Total Solids) in Water-Emulsion Waxes (**D 1289 – 55 T**),[1]
Acid Number (Empirical) of Natural Waxes (**D 1386 – 55 T**),[1] and
Saponification Number (Empirical) of Natural Waxes (**D 1387 – 55 T**).[1]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Nomenclature (J. T. Hohnstine, chairman) is preparing a glossary of terms relating to wax polishes.

Subcommittee II on Raw Materials (Melvin Fuld, chairman) is working on revisions of Tentative Methods D 1386 – 55 T and D 1387 – 55 T for the acid number and saponification number of waxes. An electrometric titration method is being investigated. Pending the completion of this work, the subcommittee recommended that these methods be continued as tentatives without change. Task groups are preparing additional test methods for various properties of natural waxes.

Subcommittee III on Chemical and Physical Testing (Gerald DeNapoli, chairman) is working on a test method for the stability of water-emulsion waxes and a method for the determination of nonvolatile matter in solvent waxes.

A task group is investigating methods for the determination of silicones in wax polishes.

Subcommittee IV on Performance Tests (C. S. Kimball, chairman) is preparing methods for testing the water spotting and removability of floor waxes. A task group is also investigating methods of determining the soil resistance of water-emulsion waxes. The subcommittee has prepared a report on the significance of slip tests on floors by the Sigler and James machines which will be published for information only in the September, 1958, ASTM BULLETIN.

Subcommittee V on Specifications (William Joy, chairman) is preparing a specification for industrial type water-emulsion maintenance practices for asphalt and mastic floors to be used as a guide for future committee work.

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* Sixty-first Annual Meeting of the Society, June 22–27, 1958.

[1] 1955 Book of ASTM Standards, Part 4.

This report has been submitted to letter ballot of the committee, which consists of 71 members; 54 members have returned their ballots, of whom 49 voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

W. W. WALTON,
Chairman.

BAYARD S. JOHNSON,
Secretary.

REPORT OF COMMITTEE D-22

ON

METHODS OF ATMOSPHERIC SAMPLING AND ANALYSIS*

Committee D-22 on Methods of Atmospheric Sampling and Analysis held one meeting during the year: on September 12, 1957, in New York City in connection with the Air Pollution Symposium of the American Chemical Society.

The officers elected for the ensuing term of two years are as follows:

Chairman, Leslie Silverman.
Vice-Chairman, J. Cholak.
Secretary, A. T. Rossano, Jr.

A Symposium on Instrumentation in Air Sampling and Analysis was held by the committee at the 1958 Annual Meeting of the Society in Boston.

NEW TENTATIVES

The committee recommends for publication as tentative the following methods and recommended practices as appended hereto:[1]

Method for:

Nitrogen Dioxide and Nitric Oxide Content of the Atmosphere (Modified Griess-Ilosvay Reaction),
Oxides of Nitrogen in Gaseous Combustion Products (Phenol–Disulfonic Acid Procedure),
Oxidant (Ozone) Content of the Atmosphere,
Inorganic Fluoride in the Atmosphere, and

Recommended Practices for:

Sampling Atmospheres for Analysis of Gases and Vapors.

REVISION OF TENTATIVES

The committee recommends revisions as indicated of the following tentatives and their continuation as tentative:

Tentative Method for Continuous Analysis and Automatic Recording of Sulfur Dioxide Content of the Atmosphere (D 1355 – 55 T):[2] Revise by adding Method B. Electrolysis Method as appended hereto.[1]

Definitions of Terms Relating to Atmospheric Sampling and Analysis (D 1356 – 55 T):[2] Revise by the addition of the definitions appended hereto.[1]

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[3]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Nomenclature and Units (F. A. Patty, chairman), prepared the revised definitions of terms (D 1356) referred to earlier in this report.

A fourteen-page list of conversion units and factors has been completed by the subcommittee and referred to the entire committee. The comments received are under consideration by the subcommittee for revision of this proposed tentative prior to obtaining a ballot on it.

Subcommittee II on Sampling (Leslie Silverman, chairman) prepared the proposed Tentative Recommended Practices for Sampling Atmospheres for

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* Sixty-first Annual Meeting of the Society, June 22–27, 1958.

[1] The new and revised tentatives appear in the 1958 Book of ASTM Standards, Part 10.

[2] 1955 Book of ASTM Standards, Part 7.

[3] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

Analysis of Gases and Vapors referred to earlier in this report. A proposed standard relating to dust fall jars is in preparation. The proposed standard for particle sizing is being referred to the subcommittee members. The proposed standard for stack sampling needs only the preparation of one table for its completion. Work is still to be done on the sampling of vegetation and soils.

Subcommittee III on Analysis (J. Cholak, chairman) prepared the method for the determination of inorganic fluoride in the atmosphere referred to earlier in the report. A method on fluorides in biological materials is being referred to the subcommittee.

Subcommittee IV on Automatic Samplers and Recorders (M. D. Thomas, chairman).—The paper filtration method for aerosol determination is ready for subcommittee ballot. Final consideration is being given to the Sinclair photometric method and limited consideration has been given to the Gucker-O'Konski method.

The oxidant recorder method for determination of ozone and other oxidants (KI reactor) has been completed.

A fluoride recorder has not been developed sufficiently to be a subject for an ASTM standard.

This report has been submitted to letter ballot of the committee, which consists of 61 voting members; 20 members returned their ballots, of whom 18 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

L. C. McCABE,
Chairman.

A. T. ROSSANO, JR.
Secretary.

REPORT OF COMMITTEE D-23
ON
CELLULOSE AND CELLULOSE DERIVATIVES*

Committee D-23 on Cellulose and Cellulose Derivatives held one meeting during the year: in New York City on September 12, 1957. The Executive Subcommittee also met in New York City on February 21, 1957.

The committee consists of 48 members, of whom 15 are classified as producers, 11 as consumers, and 22 as general interest members.

The officers elected for the ensuing term of two years are as follows:

Chairman, F. A. Simmonds.
Vice-Chairman, Kyle Ward, Jr.
Secretary, W. W. Becher.

REVISION OF TENTATIVE

The committee recommends revisions as appended hereto[1] of the Tentative Methods of Testing Sodium Carboxymethylcellulose (**D 1439 - 56 T**)[2] and continuation of the method as tentative.

REVISION OF STANDARD AND REVERSION TO TENTATIVE

The committee recommends revisions as appended hereto[1] of the Standard Method of Test for Moisture in Cellulose (**D 1348 - 56**)[2] and reversion of the method to tentative.

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[3]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Nomenclature and Definitions (Kyle Ward, Jr., chairman) has prepared a list of definitions for 117 items. Of this list 44 are satisfactory to the subcommittee and will be submitted to the entire membership of Committee D-23 for approval or comments. The remaining 73 items will be revised before such reference. A report of the subcommittee was given at the 1958 meeting of the Technical Association of the Pulp and Paper Industry and will be published in TAPPI.

Subcommittee II on Cellulose (E. S. McColley, chairman) has prepared the proposed tentative method for moisture in cellulose by the Karl Fischer method referred to earlier in this report.

Task groups on Absorption and Color, Cold Alkali Solubility, Chromatographic Methods, and Pentosans have prepared methods which are being investigated by interlaboratory testing; task groups on Disperse Viscosity and Molecular Chain Length Distribution are engaged in preparing methods; and a task group on Functional Groups is engaged in testing various procedures by a variety of methods.

Subcommittee III on Organic Esters (L. B. Genung, chairman) has furnished the Task Group on Disperse Viscosity of Subcommittee II with a practical procedure for deacylation of cellulose esters, for possible inclusion in the viscosity procedure. A method for the determination of hydroxyl in pyridine-soluble cellulose esters is available for future revisions of D 871 and D 817.

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* Sixty-first Annual Meeting of the Society, June 22–27, 1958.

[1] The revised tentative and standard appear in the 1958 Book of ASTM Standards, Part 6.

[2] 1956 Supplement to Book of ASTM Standards, Part 4.

[3] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

Subcommittee IV on Inorganic Esters (C. B. Gilbert, chairman) is preparing, in cooperation with Subcommittee XXV of Committee D-1, a single method for determination of dilution ratios of cellulose nitrate, solvents, and diluents, to replace Methods D 301 – 56, Section 16, D 268 – 49 T, and D 1134 – 53.

Subcommittee V on Cellulose Ethers (R. W. Swinehart, chairman) has prepared the proposed Tentative Methods of Testing Sodium Carboxymethylcellulose, referred to earlier in this report.

W. B. Johnson has been appointed chairman of a new Task Group on Methods for Purified Type of Sodium Carboxymethylcellulose. The present methods for moisture and viscosity in Method D 1439 – 56 T should be applicable to this type; the degree of etherification method will probably be modified. No assay (purity) procedure is contemplated.

E. P. Samsel has been appointed chairman of a task group to prepare methods for the analysis of alkyl and hydroxyalkyl ethers.

Subcommittee VI on Statistics (A. F. Johnson, chairman) has analyzed statistically the data from interlaboratory comparisons of (*a*) the Karl Fischer method for moisture in cellulose and (*b*) the assay method for sodium carboxymethylcellulose. Also, data on the alkali solubility of cellulose is being analyzed; the experimental design for this test was set up by Subcommittee VI.

An information sheet on a variance components experimental design, truncated sampling scheme, has been prepared and is available to members of Committee D-23.

A formal manual of statistical procedures was planned several years ago and several chapters were completed. Due to the availability of general statistical literature, no more work is planned; however, copies of the individual completed chapters may be obtained on request to the chairman.

This report has been submitted to letter ballot of the committee, which consists of 48 voting members; 29 members returned their ballots, of whom 27 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

F. A. SIMMONDS,
Chairman.

W. W. BECKER,
Secretary.

REPORT OF COMMITTEE D-24
ON
CARBON BLACK*

Committee D-24 on Carbon Black held two meetings during the past year: in Atlantic City, N. J., on June 20, 1957, during the Annual Meeting of the Society, and in St. Louis, Mo., on February 12, 1958, during ASTM Committee Week.

The committee consists of 44 members, of whom 11 are classified as producers, 26 as consumers, and 7 as general interest members.

The officers elected for the ensuing term of two years are as follows:

Chairman, N. P. Bekema.

Vice-Chairman, J. F. Svetlik.

Secretary, T. D. Bolt.

Executive Committee Members: I. Drogin, K. A. Burgess, C. A. Carlton, L. G. Mason, R. F. Jones, and S. R. Doner.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Physical Tests (J. F. Svetlik, chairman) is preparing a method of test for mass strength of pelleted carbon black.

Subcommittee II on Chemical Tests (L. G. Mason, chairman) has completed tentative methods of test for sulfur content and for volatile content of carbon black. An editorial correction has been made in the Tentative Method of Test for Iodine Adsorption Number of Carbon Black (ASTM Designation: D 1510 – 57 T),[1] changing the normality of the sodium thiosulfate solution from 0.394 to 0.0394.

Subcommittee III on Optical Tests (J. F. Ambrose, chairman) has completed a method of test for discoloration of benzene by carbon black.

Subcommittee V on Sampling (R. O. Treat, chairman) is preparing methods for sampling bag and bulk shipments of carbon black.

Respectfully submitted on behalf of the committee,

S. R. DONER,
Chairman.

A. G. COBBE,
Secretary.

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee D-24 presented to the Society through the Administrative Committee on Standards the following recommendations:

New Tentative Methods of Test for:

Discoloration of Benzene by Carbon Black (D 1618 – 58 T),
Sulfur Content of Carbon Black (D 1619 – 58 T), and
Volatile Content of Carbon Black (D 1620 – 58 T).

These recommendations were accepted by the Standards Committee on September 9, 1958, and the new tentative methods appear in the 1958 Book of ASTM Standards, Part 9.

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* Sixty-first Annual Meeting of the Society, June 22–27, 1958.

[1] 1957 Supplement to Book of ASTM Standards, Part 6.

REPORT OF COMMITTEE D-25

ON

CASEIN AND SIMILAR PROTEIN MATERIALS

Committee D-25 on Casein and Similar Protein Materials will complete its second full year of official existence this June. Meetings were held on June 19, 1957, in Atlantic City, N. J., and on June 18, 1958, in Lancaster, Pa. An informal discussion meeting was held on February 20, 1958, in New York City during the TAPPI Meeting.

The chairman of Subcommittee I on Definitions, R. W. Bell, resigned, and W. W. Stonebreaker was appointed to take his place. J. S. Baker was appointed as chairman of Subcommittee III on Chemical Tests following the resignation of E. G. Stimpson. E. Maynard, of the U. S. Gypsum Co., was appointed chairman of Subcommittee IV on Physical Tests following the resignation of L. E. Georgevits.

The committee consists of 21 members, of whom 6 are classified as producers, 7 as consumers, and 8 as general interest members.

The officers elected for the ensuing term of two years are as follows:

Chairman, H. W. Shader.

Vice-Chairman, L. E. Clark, Jr.

Secretary, L. E. Georgevits.

Advisory Committee Members-at-Large: C. Coriss, W. M. Bain, W. R. Willets, and Joseph Brown.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Editorial and Nomenclature (M. E. Stonebreaker, chairman).—The name of this subcommittee has been changed from "Definitions" to the above title. Rather than prepare a random list of definitions, the subcommittee will define terms as they appear in the test methods. It will also act as the editorial committee on all test methods. Methods for sampling procedure prepared by Subcommittee II have been reviewed, and test methods for determination of nitrogen and moisture are presently being reviewed.

Subcommittee II on Sampling (J. C. Rice, chairman).—Rather than go through a long series of tests on a sampling plan, it was decided to adopt one of the standard sampling procedures. The method is now being sent as a letter ballot to the full committee.

Subcommittee III on Chemical Tests (J. S. Baker, chairman).—A series of interlaboratory tests was made to determine the moisture content of a sample of casein and isolated soya protein. The proposed method was found to give good results and has been submitted to Subcommittee I for review.

Proposed methods for nitrogen determination and moisture have been prepared and are being reviewed by Subcommittee I. No cooperative tests were thought necessary.

Test methods for total ash, fixed ash, fat content, free acidity, and total acidity have been discussed. There are still some controversial points in these methods and they are being studied further.

Subcommittee IV on Physical Tests (L. E. Georgevits, chairman).—Interlaboratory tests were made to determine the particle size and viscosity of casein and isolated soya protein samples. Poor correlation was obtained and the methods for both determinations are being revised. Samples of standard viscosity oils will be sent out to check the Brookfield viscosimeters used. A method for determining the minimum alkali requirement and insoluble matter in a sample is being studied.

A method for determining adhesive strength based on preparing a paper coating and determining its strength with a standard Dennison wax is being studied.

Subcommittee VI on Research (L. E. Clark, Jr., chairman).—Various qualitative tests for distinguishing casein and isolated soya protein have been presented; one quantitative test has been suggested, but no committee work has been done on this project.

This report has been submitted to letter ballot of the committee, which consists of 21 members; 12 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

H. W. SHADER,
Chairman.

L. E. GEORGEVITS,
Secretary.

REPORT OF ALCA-ASTM JOINT COMMITTEE
ON
LEATHER*

The ALCA-ASTM Committee on Leather (joint with American Leather Chemists' Assn.) held two meetings during the year: on October 17 and 18, 1957, in Cincinnati, Ohio, and on March 27 and 28, 1958, in New York City.

The officers elected for the ensuing term of two years are as follows:

Chairman, Joseph R. Kanagy.
Vice-Chairman, B. L. Lewis.
Secretary, Charles W. Mann.

NEW TENTATIVES

The committee recommends for publication as tentative the following methods as appended hereto:[1]

Tentative Method of:

Conditioning Leather and Leather Products for Testing, and
Test for Corrosion Produced by Leather in Contact with Metal.

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[2]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Nomenclature, Definitions, and Applicability (R. M. Lollar, chairman) is reviewing test methods in regard to definitions and applicability. Particular attention is being given to statements of significance and precision of all methods passing through the committee.

Subcommittee II on Sampling and Conditioning for Physical Testing (C. W. Mann, chairman) prepared the proposed Tentative Method for Conditioning Leather and Leather Products for Testing referred to earlier in this report. Also in preparation is a method for sampling side leather; methods will also be prepared for sampling small skins.

Subcommittee III on Physical Testing (G. A. Butz, chairman):

Section 2 on Physical Dimensions (J. Naghski, chairman) is preparing a proposed method for measuring thickness.

Section 3 on Tensile Properties (R. L. Young, chairman) is working on methods for tensile strength; stitch tear, double hole; and breaking strength; small ball burst strength; and elongation.

Section 4 on Measurement of Water Resistance (R. G. Ashcraft, chairman) has prepared a method for static water absorption which awaits further information on significance and precision. A method for dynamic water penetration resistance of upper leathers has been prepared and awaits further data on significance and precision. Work is under way on methods for spray testing of garment leather, dynamic water absorption of light leathers, and penetration resistance of glove leathers.

Section 5 on Surface Characteristics (Robert Stubbings, chairman) is preparing a method for cold flex testing and one for the flex testing of finish on upholstery leathers. Also being studied are methods to evaluate crocking, flex testing of finish on side upper leather, a

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*** Sixty-first Annual Meeting of the Society, June 22–27, 1958.**

[1] The new tentatives appear in the 1958 Book of ASTM Standards, Part 6.

[2] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

light fastness test for leather, and a scuff resistance test for leather.

Section 7 on Mechanical Leathers (Miscellaneous Properties) (A. N. Compton, chairman) prepared the proposed Tentative Method of Test for Corrosion Produced by Leather in Contact with Metals and is working on a method for oil and air permeability of leather cup packings.

Section 8 on Deterioration Effects (S. S. Kremen, chairman) is working on methods to evaluate the perspiration resistance of insole leather and white upper leathers.

Section 10 on Miscellaneous Physical Properties (A. N. Kay, chairman) is working on methods to determine the following characteristics of leather: pipe, stiffness, and compressibility.

Subcommittee IV on Research (M. M. Baldwin, chairman) is compiling a list of current research on test methods both within the committee and by the leather industry in general. This committee is recommending to the Advisory Committee areas for research on new test methods and is preparing for ASTM a list of suggested research projects which might be carried out by students.

Subcommittee V on Editorial (T. Thorstensen, chairman) has concerned itself principally with the proper form and organization for presentation of test methods.

This report has been submitted to letter ballot of the committee which consists of 66 members; 54 members returned their ballots, of whom 32 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

J. R. KANAGY,
Chairman.

M. H. BATTLES,
Secretary.

REPORT OF COMMITTEE E-1
ON
METHODS OF TESTING*

Committee E-1 on Methods of Testing held two meetings during the year: at Hoboken, N. J., on December 13, 1957; and at Philadelphia, Pa., on April 17, 1958. Seventeen subcommittees and tasks groups met during the 1957 Annual Meeting of the Society.

Meetings were held of fourteen E-1 subcommittees and task groups during ASTM Committee Week, in St. Louis, Mo., from February 9 to 14, 1958.

Meetings of the following subcommittees were held in New York, N. Y., on October 24 and 25, 1957: Subcommittee 17 on Thermometers, Subcommittee 18 on Hydrometers, and Subcommittee 21 on Metalware Laboratory Apparatus.

Mr. J. R. Townsend found it necessary to resign as Chairman of Committee E-1 because of his many duties as Special Assistant in the Office of the Assistant Secretary of Defense. In accepting Mr. Townsend's resignation, the committee adopted a resolution expressing the best wishes of the committee in his new assignment, and recorded with appreciation the many contributions and services which Mr. Townsend made to the work of Committee E-1 during his long association with it and especially during the past ten years as its chairman.

At the April 17 meeting, the following officers were elected for the ensuing term of two years:

Chairman, A. C. Webber, and
Vice-Chairman, D. E. Parsons.

Subcommittee 23 on Volatility has been discontinued until such time as there is an indicated need for again undertaking active work on this subject.

Task Groups F and I have been established as permanent subcommittees, namely, Subcommittee 30 on Bend Testing, and Subcommittee 31 on Laboratory Balances.

Committee E-1 is sponsoring a Symposium on Particle Size Measurement[1] during the 1958 Annual Meeting of the Society. This Symposium which comprises fifteen papers will be held in three sessions on June 26 and 27.

The Instrument and Apparatus Industry Luncheon at the Annual Meeting on Thursday, June 26, is being sponsored by Committee E-1.

Plans are under way for a Symposium on Methods and Techniques of Light and Electron Microscopy to be held at the 1959 Annual Meeting under the auspices of Subcommittee 28 on Microscopy. Mr. F. F. Morehead, American Viscose Co., is chairman of the Symposium Committee.

Consideration is being given to undertaking work on a thermal conductivity test for metals, provided there is sufficient interest among other Society committees to collaborate on this project.

A Survey Committee on Bimetallic Thermometers (R. D. Thompson, chairman) reported that while there appears to be some interest in this subject, it is not sufficient to justify further study or the creation of a task group at this time.

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* Sixty-first Annual Meeting of the Society, June 22–27, 1958.

[1] To be issued as separate publication *ASTM STP No. 234*.

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1957 Annual Meeting, Committee E-1 presented to the Society through the Administrative Committee on Standards the following recommendations:

Revision of Tentative Methods of:

Test for Rockwell Hardness and Rockwell Superficial Hardness of Metallic Materials (E 18 - 55 T), and

Tension Testing of Metallic Materials (E 8 - 54 T).

Revision of Tentative Definitions:

With Procedures Relating to Conditioning and Weathering (E 41 - 48 T).

Revision of Tentative Specifications for:

Weighing and Drying Apparatus for Microchemical Analysis (E 124 - 56 T).

The revision of Tentative Methods E 18 - 55 T was accepted by the Standards Committee on November 1, 1957 and appears in the 1957 Supplement to Book of ASTM Standards, Parts 1 and 2. The revision of Tentative Methods E 8 - 54 T was accepted on December 26, 1957 and appears in Part 1 of the 1957 Supplement. The revision of Tentative Definitions E 41 - 48 T was accepted on December 23, 1957 and is set forth on yellow stickers provided with Parts 3, 4, 5, 6 and 7 of the 1957 Supplement. The revision of Tentative Specifications E 124 - 56 T was accepted on December 23, 1957 and appears in Parts 3, 4 and 5 of the 1957 Supplement.

NEW STANDARD, IMMEDIATE ADOPTION

The committee recommends that the Specifications for Distillation Equipment, as appended hereto,[2] be approved for reference to letter ballot of the Society for adoption as standard, and accordingly requests the necessary nine-tenths affirmative vote at the Annual Meeting in order that the specifications may be referred to letter ballot of the Society.

These specifications had been published as information in an Appendix to the 1957 Report; they cover distillation equipment used in six ASTM methods of test. They were prepared jointly by Subcommittee 19 on Glassware Laboratory Apparatus and Subcommittee 21 on Metalware Laboratory Apparatus.

NEW TENTATIVES

The committee recommends for publication as tentative the following specifications and methods, as appended hereto:[2]

Specifications for Pensky-Martens Closed Flash Tester, and

Method for Determination of Poisson's Ratio at Room Temperature.

REVISION OF TENTATIVES

The committee recommends revisions, as indicated, of the following tentatives and their continuation as tentative:

Definition of Terms Relating to Methods of Mechanical Testing (E 6 - 54 T):[3] Revise as appended hereto:[2,4]

In addition to a number of changes in the present definitions, there have been added definitions of terms covering fatigue and bend tests.

Specifications for Cell-Type Oven with Controlled Rates of Ventilation (E 95 - 52 T):[3]

Section 2.—Add the following sentence to Paragraph (*a*): "The cells shall not be constructed of copper or a copper alloy."

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[2] The new and revised tentatives and standards appear in the 1958 Book of ASTM Standards.

[3] 1955 Book of ASTM Standards.

[4] See Editorial Note, p. 442.

TABLE I.—PROPOSED CHANGES IN PRECISION THERMOMETER SPECIFICATIONS.

ASTM Thermometer No.	62C	62F	63C	63F	64C	64F
Temperature Range	−38 to +2 C	−36 to +35 F	−8 to +32 C	18 to 89 F	25 to 55 C	77 to 131 F
Total Length	374 to 384 mm		374 to 384 mm		374 to 384 mm	
Bottom of Bulb to Graduation Line at	−35 C	−31 F	−5 C	23 F	25 C	77 F
Distance	77 to 98 mm		77 to 98 mm		115 to 135 mm	
Bottom of Bulb to Graduation Line at	0 C	+32 F	+30 C	86 F	55 C	131 F
Distance	307 to 330 mm		307 to 330 mm		324 to 344 mm	
Expansion Chamber shall permit heating to	55 C	130 F	80 C	175 F	105 C	220 F

ASTM Thermometer No.	65C	65F	66C	66F	67C	67F
Temperature Range	50 to 80 C	122 to 176 F	75 to 105 C	167 to 221 F	95 to 155 C	203 to 311 F
Total Length	374 to 384 mm		374 to 384 mm		374 to 384 mm	
Bottom of Bulb to Graduation Line at	50 C	122 F	75 C	167 F	95 C	203 F
Distance	115 to 135 mm		115 to 135 mm		115 to 135 mm	
Bottom of Bulb to Graduation Line at	80 C	176 F	105 C	221 F	155 C	311 F
Distance	324 to 344 mm		324 to 344 mm		324 to 344 mm	
Expansion Chamber shall permit heating to	130 C	265 F	155 C	310 F	180 C	355 F

ASTM Thermometer No.	68C	68F	69C	69F	70C	70F
Temperature Range	145 to 205 C	293 to 401 F	195 to 305 C	383 to 581 F	295 to 405 C	563 to 761 F
Total Length	374 to 384 mm		374 to 384 mm		374 to 384 mm	
Bottom of Bulb to Graduation Line at	145 C	293 F	195 C	383 F	295 C	563 F
Distance	115 to 135 mm		115 to 135 mm		115 to 135 mm	
Bottom of Bulb to Graduation Line at	205 C	401 F	305 C	581 F	405 C	761 F
Distance	324 to 344 mm		324 to 344 mm		324 to 344 mm	
Expansion Chamber shall permit heating to	230 C	445 F	330 C	625 F	[a]	[a]

[a] The expansion chamber is provided for relief of gas pressure to avoid distortion of the bulb at higher temperatures. It is not for the purpose of joining mercury separations, and under no circumstances should the thermometer be heated above the highest temperature reading.

Change the first sentence of Paragraph (*b*) to read: "The design of the oven shall be such that heated air enters one end of the cell, and is exhausted from the other end of it without being recirculated." Add the following sentence at the end of Paragraph (*b*): "The design shall also permit the cells to be cleaned easily after each test."

Method for Determination of Young's Modulus at Room Temperature (E 111 - 55 T):[3]

Section 1.—Add the following sentence at the end of this section: "The method is limited to materials in which creep is negligible compared to the strain produced immediately upon loading."

REVISION OF STANDARDS, IMMEDIATE ADOPTION

The committee recommends for immediate adoption revisions as indicated of the following four standards and accordingly asks for the necessary nine-tenths affirmative vote at the Annual Meeting in order that the revisions may be referred to letter ballot of the Society:

Standard Specifications for ASTM Thermometers (E 1 - 57):[5]

Section 2.—Add the following at the end of Note 1: "The expansion chamber is an enlargement at the top end of the capillary bore which shall have a capacity equivalent to not less than 20 mm of unchanged capillary. Any enlargement at the top end of the capillary bore smaller than this is not to be regarded as an expansion chamber."

Glass Ring.—Add requirements for the glass ring, that, when specified, the outside diameter of the glass ring shall be not more than the diameter of the stem, and the inside diameter not less than 2 mm.

Thermometers 1C, 1F, 2C, 2F, 3C, 3F.—Revise the requirements for the three general ASTM partial immersion thermometers as shown in the accompanying Table II.

Thermometers 2C, 2F, 7C and 7F.—Add the following requirement for expansion chamber: "Expansion chamber shall permit heating to not over 400 C or 760 F."

TABLE II.—PROPOSED CHANGES IN GENERAL PARTIAL IMMERSION THERMOMETER SPECIFICATIONS.

ASTM Thermometer No.	1C	1F	2C	2F	3C	3F
Total Length	317 to 327 mm		385 to 395 mm		408 to 418 mm	
Bottom of Bulb to Graduation Line at	150 C	302 F	300 C	752 F	49 C	120 F
Distance	268 to 287 mm		329 to 358 mm		354 to 383 mm	

Thermometers 3C, 3F, 8C, 8F, 10C, 10F, 11C, 11F, 70C and 70F.—Add a footnote to read as follows:

> "The expansion chamber is provided for relief of gas pressure to avoid distortion of the bulb at higher temperatures. It is not for the purpose of joining mercury separations; and under no circumstances should the thermometer be heated above the highest temperature reading."

Precision Thermometers.—Revise the requirements for precision thermometers 62 C to 70 F as shown in the accompanying Table I.

Hardness Conversion Table for Cartridge Brass (E 33 - 42),[3] for Steel (E 48 - 47),[3] and for Nickel and High-Nickel Alloys (E 93 - 52):[3]

The committee recommends that these three hardness conversion tables be combined and the text revised in accordance with the Standard Hardness Conversion Tables for Metals (Relationship Between Brinell Hardness, Diamond Pyramid Hardness, Rockwell Hardness, and Rockwell Superficial Hardness), as appended hereto.[2]

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[5] 1957 Supplement to Book of ASTM Standards.

ADOPTION OF TENTATIVES AS STANDARD WITH REVISION

The committee recommends that the following two tentatives be approved for reference to letter ballot of the Society for adoption of standard with revisions as indicated:

Specifications for ASTM Thermometers (E 1 – 57):[5]

Adoption as standard of the following tentative thermometer specifications:

Name	ASTM Thermometer No.
Aniline Point	33C, 33F, 34C, 34F, 35C, 35F
Butadiene Boiling Point Range	52C
Antifreeze Freezing Point	75F, 76F
Saybolt Viscosity	77F, 78F, 79F, 80F, 81F
Fuel Rating	82F, 83F, 84F, 85F, 86F, 87F
Tank	97F, 98F

Method of Testing and Standardization of Etched-Stem Liquid-in-Glass Thermometers (E 77 – 56 T):[6]

Section 4 (*d*).—After the first sentence, add a new sentence to read, "A magnifier of 10 × is recommended for visual examination."

TENTATIVES CONTINUED WITHOUT REVISION

The committee recommends continuation without revision of the remaining tentatives under its jurisdiction.

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[7]

ACTIVITIES OF SUBCOMMITTEES AND TASK GROUPS

Subcommittee 1 on Calibration of Mechanical Testing Machines and Apparatus (B. L. Wilson, chairman).—Editorial changes in the Tentative Methods of Verification of Testing Machines (E 4 – 57 T) are under consideration. Editorial changes in the Tentative Method of Verification and Classification of Extensometers (E 83 – 57 T) are also in preparation to emphasize that the method is not a complete purchase specification, and also to include a statement that all ranges of multi-range extensometers should be verified and classified. At the request of Committee D-11 on Rubber and Rubber-Like Materials, consideration is being given to methods for calibrating testing machines used for testing rubber and textiles.

Subcommittee 2 on Effect of Speed in Mechanical Testing (W. Ramberg, chairman).—This subcommittee has been considering new developments in apparatus for controlling the speed of testing machines, including equipment for manual or automatic control of strain pacing, load pacing, and crosshead pacing over a wide range. Attention is being given to present-day needs for dual range extensometers with high magnification in the elastic range and low magnification in the plastic range to be used in tests at controlled speeds.

Subcommittee 3 on Elastic Strength of Materials (G. R. Gohn, chairman).—Subcommittee 3 completed the revisions in the Tentative Method for the Determination of Young's Modulus at Room Temperature (E 111 – 55 T) and in the Tentative Definitions of Terms Relating to Methods of Mechanical

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[6] 1956 Supplement to Book of ASTM Standards, Part 5.

[7] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

Testing (E 6 – 54 T). New definitions pertaining to fatigue and bend testing also have been included in the proposed revision of Definitions E 6. A Task Group under H. L. Fry is reviewing terms pertaining to creep and relaxation testing for possible inclusion.

A Task Group under Walter Ramberg developed the proposed Tentative Method for Determination of Poisson's Ratio at Room Temperature. It is now engaged in the preparation of a test method for the determination of the shear modulus in torsion at room temperature.

Subcommittee 4 on Tension Testing (F. M. Howell, chairman).—A number of important changes in the Tentative Methods of Tension Testing of Metallic Materials (E 8 – 57 T) were made last year. Because further changes are now under consideration, these methods are being retained as tentative. Consideration is now being given to the preparation of general methods of tension testing of non-metallic materials.

Subcommittee 6 on Indentation Hardness (R. H. Heyer, chairman).—The new Standard Hardness Conversion Tables for Metals will simplify the addition of new conversion tables as needed and replace the three existing hardness conversion tables for Cartridge Brass (E 33), Steel (E 48), and Nickel and High-Nickel Alloys (E 93).

The Task Group on Hardness Conversions has under consideration conversion tables for copper-beryllium alloy strip. Hardness conversion tables for aluminum alloys are being studied.

The Joint Committee for Steel Hardness-Tensile Conversions, functioning as a Task Group of this subcommittee, is making excellent progress in its round-robin test program to develop data to check hardness-tensile conversions for steel at high-strength levels.

The Task Group on Brinell Hardness has prepared a rather extensive revision of Methods E 10 – 54 T which is about to be submitted to letter ballot of the subcommittee.[4]

The Task Group on Diamond Pyramid Hardness is studying errors in applying the load in certain weight-lever type instruments.

The Task Group on Rockwell Hardness is studying corrections for rounds in tests using ball indentors.

Subcommittee 9 on Rheological Properties (W. F. Fair, Jr., chairman).—The Task Group on Saybolt Furol High-Temperature Viscosimeter (R. R. Thurston, chairman) completed its work and has been discharged.

Task Group on Definitions (R. N. Traxler, chairman) has completed a revision of the Standard Definition of Terms Relating to Rheological Properties of Matter (E 24 – 42). These revised definitions will be submitted to the Society through the Administrative Committee on Standards later this year.[4]

Task Group on Softening Point (M. D. Chamberlain, chairman) is considering a procedure for sample preparation (by molding) of heat-sensitive resins prepared by Committee D-1 on Paint, Varnish, Lacquer, and Related Products for inclusion in the Tentative Method of Test for Softening Point by Ring-and-Ball Apparatus (E 28 – 51 T). This method will be an alternate procedure to the pour method and the powder method. It is now used routinely in some laboratories and has the advantage over the powder method of requiring only simple laboratory equipment. This revision of Method E 28 will be presented to the Society through the Administrative Committee on Standards after the Annual Meeting.[4]

Subcommittee 12 on Methods for Density (C. T. Collett, chairman) is considering a proposed Method of Test for Bulk Density of Abrasive Grain which was submitted to the Society by the Abrasive Grain Assn.

Subcommittee 14 on Conditioning and Weathering (A. C. Webber, chairman).—The chairman attended the meeting in Paris of the ISO Coordinating Committee on Atmospheres for Conditioning and Testing (ATCO). This advisory group recommended to the General Council of ISO that each ISO Technical committee, for which conditioning was of importance, select its standard atmospheres from the following:

20 ± 2 C and 65 ± 5 per cent RH
23 ± 2 C and 50 ± 5 per cent RH
27 ± 2 C and 65 ± 5 per cent RH

The same tolerances were recommended for each condition, with the provision that closer tolerances (1 C and 2 per cent RH) may be applied where required. The addition of these conditions, while not providing the single standard hoped for, recognizes the existence and extensive use of each of the conditions and should prevent the adoption of other atmospheres for normal testing.

The Tentative Definitions with Procedures Relating to Conditioning and Weathering (E 41 – 48 T), were reviewed. While extensive revisions are required, an immediate ballot was taken to change the temperature tolerance from ±1.1 C (2 F) to ±1 C (1.8 F). This recommendation was adopted in 1957.

The committee discussed the need for an E Method covering all methods for measurement of Relative Humidity. Method D 337, under the jurisdiction of Committee D-13 on Textile Materials was referred to E-1 Subcommittee 14 for revision and publication as a general method.

Consideration is being given to a proposed revision of the Tentative Methods of Test for Measuring Water Vapor Transmission of Materials in Sheet Form (E 96 – 53 T).

Subcommittee 16 on Thickness Measurement (S. B. Newman, chairman).—The Method of Test for Thickness of Electrical Insulation (D 374 – 57 T) was revised and reverted to tentative in 1957. A revised version with increased scope is now being prepared with a view to its recommendation for adoption as standard.

Subcommittee 17 on Thermometers (R. M. Wilhelm, chairman) submitted the revisions in the Specifications for ASTM Thermometers (E 1 – 57) and also recommended the adoption as standard of the Tentative Method of Testing and Standardization of Etched-Stem Liquid-in-Glass Thermometers (E 77 – 56 T). One of the principal changes in the thermometer specifications is a change in the method of dimensioning which applies to the group of precision thermometers. A similar change in the method of dimensioning is also under consideration for all of the other thermometers in Standard Specifications E-1. This important revision involves (1) specifying all dimensions from the bottom of the bulb without changing the range span or the location from the present values, (2) increase in the over-all length as necessary to provide proper capillary clearances at the top, and (3) changes in the over-all length tolerances to facilitate manufacture.

A paper describing studies of "Thermal Stability of Glasses in ASTM Thermometers" by E. L. Ruh and G. J. Conklin is being submitted for consideration for publication in the ASTM BULLETIN.[8]

Subcommittee 17 recommended to Committee E-1 that the Society adopt the practice of using the designation "Celsius (Centigrade)" in place of "Centigrade." A brief discussion of this, prepared by R. D. Thompson, Secretary of Subcommittee 17, will appear in an early issue of the ASTM BULLETIN.[9]

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[8] ASTM BULLETIN, No. 233, Oct., 1958, p. 35 (TP 217).

Subcommittee 18 on Hydrometers (R. D. Thompson, chairman).—An important accomplishment has been the completion of the Method for Inspection and Verification of Hydrometers (E 126 - 57 T) published last year as tentative.

Efforts at reconciliation of ASTM and API hydrometer specifications have borne fruit with the adoption by the Society of certain dimensional changes which are incorporated in the Specifications for ASTM Hydrometers (E 100 - 57 T). The problem of thermometer ranges for thermo-hydrometers is still under study by the subcommittee.

The subcommittee has actively participated in the work of ISO/TC 48 on Laboratory Glassware and Related Apparatus in developing specifications for specific gravity hydrometers as international standards. This action necessitates changes in certain of the ASTM specifications[4] which have been approved by the subcommittee subject to confirmation by Committee D-2 on Petroleum Products and Lubricants and Committee D-15 on Engine Antifreezes.

A change in the Methods of Test for Specific Gravity of Industrial Aromatic Hydrocarbons (D 891 - 51) has been recommended to Committee D-16 on Industrial Aromatic Hydrocarbons concerning the hydrometer specifications in that method.

Additional subjects under study are methods for testing hydrometers for low-surface tension liquids with specific gravities above 1.070, readjustment of tolerances for API short-form hydrometers 21H to 40H, and the need for some of the hydrometers in this series.[4]

Subcommittee 19 on Glassware Laboratory Apparatus (J. J. Moran, chairman) prepared the proposed Standard Specifications for Distillation Equipment. A number of new projects have been received in the last few months and will be considered at a meeting to be held during the Annual Meeting.

Subcommittee 21 on Metalware Laboratory Apparatus (E. L. Ruh, chairman) prepared the Tentative Specifications for Pensky-Martens Closed Flash Tester in cooperation with Committee D-2 on Petroleum Products. These specifications will be a companion standard to ASTM Method D 93. The proposed Standard Specifications for Distillation Equipment were prepared jointly with Subcommittee 19 and Committee D-2. These requirements for distillation apparatus will supplement the composite procedure in ASTM Method D 86.

Safety requirements proposed by the task group for oxygen and peroxide bombs have been referred to Committee D-2 for consideration. A similar request for assistance for rubber aging bomb operation received from Committee D-11 on Rubber Products is now under consideration.

Subcommittee 25 on Shear and Torsion Tests (R. W. Fenn, Jr., chairman) is undertaking a review of requirements for shear and torsion tests for the following materials: (1) Ceramic type materials, (2) metals and wood, (3) plastics and elastomers, (4) sandwich construction, spot welded, riveted, and adhesive-bonded joints.

Subcommittee 27 on Low-Temperature Testing of Elastomers and Plastics (R. S. Havenhill, chairman).—The fourth phase of this subcommittee's work has been completed and is published in a paper on "Brittleness Testing of Elastomers and Plastics," by A. C. Webber in the January, 1958, ASTM BULLETIN. The final phase of this work will be a report now in preparation comparing the hardness, stiffness, brittleness, and rheological tests and making recommendations regarding their use. A report on the results of laboratory tests of rheological creep of natural and GR-S rubbers is also in preparation.

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[9] ASTM BULLETIN, No. 230, May, 1958, p. 10.

Mathematical equations are being studied for the conversion of Shore A and Rex Hardness *versus* Olsen, and Pusey & Jones instruments. These equations will take into account variations in creep produced by variation in testing temperatures, stocks, and other factors.

Subcommittee 28 on Microscopy (F. G. Foster, chairman).—This subcommittee is now completing proposed Specifications and Methods of Tests for Glass Covers and Slides used in Microscopy and Specifications for Immersion Fluids for Use in Microscopy. After approval by committee letter ballot these will be submitted to the Society through the Administrative Committee on Standards. A list of definitions of terms used in microscopy is under review. A Symposium on Microscopy is being planned for the 1959 Annual Meeting.

Subcommittee 29 on Microchemical Apparatus (A. Steyermark, chairman). —This subcommittee has had three meetings at which apparatus used in connection with the determinations of nitrogen by the micro-Kjeldahl and micro-Dumas methods and for the micro-carbon-hydrogen test were studied. The work on the micro-Kjeldahl apparatus has been completed and is being prepared for submission to the Society. The work on the micro-Dumas apparatus has progressed to the point where recommendations will be able to be made in the near future. Consideration will next be given to micro-volumetric glassware.

Subcommittee 30 on Bend Testing (W. H. Mayo, chairman).—The former Task Group F has now been established as a subcommittee. Proposed definitions for bend test were submitted to Subcommittee 3 and are incorporated in the current revision of Definitions E 6.

A guided bend test designed primarily for use in testing welds, although it is applicable to bend testing of non-welded material, is now under study. This method has been drafted to be as consistent as feasible with the provisions of the pertinent sections of the ASME Boiler Construction Code, API, and AWS Codes.

The committee is considering a method covering the general area of bend testing outside of the field involving welding. It will be patterned after the Federal Test Methods Standards No. 151 for Metals, and the ISO drafts of specifications for bend testing of steel products.

Subcommittee 31 on Laboratory Balances (L. B. Macurdy, chairman).—The former Task Group I has now been established as a subcommittee. It has under consideration a series of definitions of terms relating to balance and balance performance. It has prepared a classification of balances and is considering the critical and significant concepts related to the performance of balances and the development of appropriate test methods.

This report has been submitted to letter ballot of the committee, which consists of 50 voting members; 36 members returned their ballots, of whom 35 have voted affirmatively and 1 negatively.

Respectfully submitted on behalf of the committee,

A. C. WEBBER,
Acting Chairman.

P. J. SMITH,
Secretary.

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee E-1 presented to the Society through the Administrative Committee on Standards the following recommendations:

Revision of Tentative Specifications for:

ASTM Hydrometers (E 100 – 57 T).

Revision of Tentative Method of Test for:

Brinell Hardness of Metallic Materials (E 10 – 54 T), and
Softening Point by Ring and Ball Apparatus (E 28 – 51 T).

Revision of Tentative Definitions of Terms Relating to:

Methods of Mechanical Testing (E 6 – 58 T).

Revision and Reversion to Tentative of Standard Definitions of Terms Relating to:

Rheological Properties of Matter (E 24 – 42).

Revision and Reversion to Tentative of Standard Specifications for:

Sieves for Testing Purposes (Wire Cloth Sieves, Round-Hole and Square-Hole Screens or Sieves) (E 11 – 39).

The revisions of E 28 and E 100 were accepted by the Standards Committee on October 20, 1958, and the remaining recommendations were accepted on September 9, 1958. All appear in the 1958 Book of ASTM Standards.

REPORT OF COMMITTEE E-2
ON
EMISSION SPECTROSCOPY*

Committee E-2 on Emission Spectroscopy held two meetings during the year: in Atlantic City, N. J., on June 19, 1957, and in Pittsburgh, Pa., on March 6, 1958. The Executive Subcommittee met at these times as well as in New York, N. Y., on November 8, 1957.

The committee prepared a revised edition of the book entitled "Methods for Emission Spectrochemical Analysis" which was published in November, 1957.

A symposium on Spectrographic Analysis for Trace Elements was sponsored by the committee at the 1957 Annual Meeting of the Society in June. The six papers relate to the present status of determination of gases in metals, and trace elements in metals, plants, soils, minerals, and geological materials. They were published as *ASTM STP No. 221* in May 1958.

A symposium on Spectroscopic Light Sources is being planned for the 1959 Annual Meeting of the Society.

The committee consists of 200 members. This represents an increase of 21 members since the last annual meeting.

The officers elected for the ensuing term of two years are as follows:

Chairman, D. L. Fry.
Vice-Chairman, R. E. Michaelis.
Secretary, Cyrus Feldman.

SUGGESTED METHOD TO BE PUBLISHED AS INFORMATION

The committee recommends for publication as information only the Suggested Method for Spectrochemical Analysis of Caustic Soda by the Solution - A-C Arc Technique, as appended hereto.[1]

NEW TENTATIVE

The committee recommends for publication as tentative the Definitions for Terms Relating to Emission Spectroscopy, as appended hereto.[2]

TENTATIVES CONTINUED WITHOUT REVISION

The committee recommends the continuation without revision of the following tentatives:

Tentative Recommended Practice for:

Photographic Processing in Spectrochemical Analysis (**E 115 - 56 T**), and
Photographic Photometry in Spectrochemical Analysis (**E 116 - 56 T**).

Tentative Method for Spectrochemical Analysis of:

Zinc-Base Alloys and High-Grade Zinc by the Solution - D-C Arc Technique (**E 27 - 53 T**),
Tin Alloys for Minor Constituents and Impurities (**E 51 - 43 T**),
Aluminum and Aluminum-Base Alloys by the Point-to-Plane Spark Technique (**E 101 - 53 T**), and
Pig Lead by the Point-to-Plane Spark Technique (**E 117 - 56 T**).

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[3]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Apparatus and Equipment (A. J. Mitteldorf, chairman) is compiling a summary of the characteristics of the various emulsions used throughout the world. This group is also making plans to test cut film of SA No. 1 emulsion, and is preparing a report comparing specifications of the various modern spectrographs available in the United States and Canada.

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* Sixty-first Annual Meeting of the Society, June 22-27, 1958.

[1] See p. 446.

[2] The new tentative appears in the 1958 Book of ASTM Standards, Part 7.

[3] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

Subcommittee II on Fundamental Practices (R. W. Smith, chairman) is preparing a suggested practice for the computation of the precision and accuracy of spectrographic data. The subcommittee is developing a suggested recommended basic practice in flame photometry and is assembling a bibliography on flame photometry suitable for publication by the Society. A program is in progress on testing powder specimens to determine what information should be included in a standard method of analysis by X-ray fluorescence techniques. The subcommittee is also compiling a bibliography on X-ray fluorescence and is making a direct personal appeal to the authors of outstanding papers to write suggested methods for X-ray fluorescence. A further task in progress is the compilation of many unsolved problems in spectrochemical analysis.

Subcommittee III, Editorial (E. B. Owens, chairman) has prepared final copy for the Regulations for ASTM Committee E-2 on Emission Spectroscopy and Recommendations for Preparation of Spectrochemical Methods and has submitted it to ASTM headquarters for publication. This subcommittee is continually reviewing proposed recommendations for form of methods and recommending changes in the E-2 forms to keep in step with the latest ASTM procedures. During the past year assistance was given in the publication of three items: (1) the 1957 Annual Report of Committee E-2, containing thirteen methods, reports, and practices; (2) the 1957 Revision of Methods for Emission Spectrochemical Analysis, containing thirty-two items not in the previous edition; and (3) the Symposium on Spectrochemical Analysis for Trace Elements, *ASTM STP No. 221.*

The subcommittee proposed recommended tentative definitions of 27 terms, of which 23 were accepted by the committee.[2] These definitions have grown out of the Suggested Nomenclature in Applied Spectroscopy (SM 3-1), which appears in "Methods for Emission Spectrochemical Analysis." The subcommittee is working on 30 more definitions.

Subcommittee IV on Electrodes, Pure Materials, Reagents, and Standards (W. J. Edgar, chairman) is maintaining a current list of all standards and pure materials available throughout the world and is preparing a revision of the Tentative Recommended Practice for Designation of Shapes and Sizes of Preformed Graphite Electrodes (E 130 – 57 T).

Subcommittee V on Copper, Nickel, and Their Alloys (A. W. Young, chairman) is assisting the National Bureau of Standards in preparing copper-alloy standard samples and is reviewing Suggested Method E-2 SM 5-2 (Spectrochemical Analysis of Wrought Copper Alloys by the D-C Arc Technique) with a view toward establishing it as a tentative. The subcommittee is preparing suggested methods pertaining to the analysis of nickel-base alloys.

Subcommittee VI on Lead, Tin, Zinc, and Related Materials (G. L. Crumrine, chairman) is preparing to test cooperatively E-2 SM 6-15 (Zinc Die-Casting Metal and High-Purity Zinc by the Cast Pin and Point-to-Plane Techniques) to determine whether the method can be published as tentative. Two new suggested methods, one pertaining to the analysis of zinc, are being reviewed.

Subcommittee VII on Aluminum, Magnesium, and Their Alloys (T. M. Hess, chairman) is cooperatively testing the Suggested Method for Spectrochemical Analysis of Magnesium-Base Alloys by the Point-to-Plane Spark Technique (E-2 SM 7-4). A new suggested method for the spectrochemical analysis of magnesium alloys for calcium by the flame photometer technique is being cooperatively tested by five laboratories. A new suggested method for the analysis of magnesium-base alloys is also being reviewed.

Subcommittee VIII on Titanium, Zirconium, and Related Metals (N. E. Gordon, chairman) is considering several suggested methods for the analysis of zirconium alloys and is working with the Materials Branch of the Atomic Energy Commission and the National Bureau of Standards to obtain zirconium and Zircaloy standards.

Subcommittee IX on Ferrous Alloys (J. F. Woodruff, chairman) is setting up cooperative test programs for seven different methods prior to their submittal as tentatives. The seven methods are as follows:

1. Plain Carbon and Low-Alloy Steel by the Point-to-Plane Technique,
2. Plain Carbon and Low-Alloy Steel by the Rod-to-Rod Technique,
3. Carbon and Low-Alloy Steels by the Pellet Technique,
4. Stainless-Type Steels by the Point-to-Plane Spark-Initiated Intermittent Arc Technique Using a Recording Photoelectric Spectrometer,
5. Stainless-Type Steels by the Point-to-Plane Spark Technique Using a Clock Indicating Photoelectric Spectrometer,
6. Determination of Acid-Soluble Aluminum in Low-Alloy Steels by the Solution – D-C Arc Technique, and
7. Steel for Boron by the Point-to-Plane Intermittent Arc Technique.

The subcommittee is also reviewing two new suggested methods.

Subcommittee X on Glass, Ceramics, Alkalies, and Cementitious Materials (E. S. Hodge, chairman) prepared the Suggested Method for Spectrochemical Analysis of Caustic Soda by the Solution - A-C Arc Technique mentioned earlier in this report. A suggested method for the analysis of caustic soda and brine as sodium chloride, and a suggested method for the analysis of glass for the alkaline earth elements are being written.

Subcommittee XI on Slags, Ores, and Other Miscellaneous Nonmetallic Materials (R. G. Russell, chairman) is reviewing the following four suggested methods for the spectrochemical analysis of: (1) Blast Furnace and Steel Making Slags; (2) Miscellaneous Powder Samples by the D-C Arc Technique; (3) Cerium Minerals for the Rare Earth Elements and Thorium by the D-C Arc Technique, and (4) Titanium Oxide by the D-C Arc Technique.

This report has been submitted to letter ballot of the committee, which consists of 200 members; 119 members returned their ballots, of whom 115 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

DAVID L. FRY,
Chairman.

CYRUS FELDMAN,
Secretary.

APPENDIX

SUGGESTED METHOD FOR SPECTROCHEMICAL ANALYSIS OF CAUSTIC SODA BY THE SOLUTION - A-C ARC TECHNIQUE[1]

SUBMITTED BY TASK GROUP 1 OF SUBCOMMITTEE X[2]

E-2 SM 10-15

Scope

1. This method provides for the determination of fourteen impurity elements in caustic soda. The concentration ranges covered on the basis of anhydrous caustic soda are as follows:

Element	Concentration Range, ppm
Silicon	4 to 300
Strontium	4 to 150
Calcium	2 to 150
Aluminum	0.8 to 150
Iron	4 to 75
Nickel	1.5 to 75
Chromium	4 to 40
Lead	4 to 40
Vanadium	2 to 40
Boron	0.4 to 40
Cobalt	0.4 to 40
Magnesium	0.4 to 40
Copper	0.1 to 40
Manganese	0.1 to 40

Summary of Method

2. The basis of this procedure has been reported in the literature.[3] Samples are dissolved or diluted in distilled water. To a known weight of sodium hydroxide a measured volume of an internal standard solution of sodium molybdate is added. The solution is then diluted to provide a 25 per cent solution of sodium hydroxide. A drop of the solution is applied to each of a pair of pure graphite electrodes and is dried by suitable means in an atmosphere of carbon dioxide. The electrode pair is excited in an a-c arc, and the spectrum is recorded photographically. Intensity ratios of analytical lines and internal standard molybdenum lines are determined photometrically, and elemental concentrations are read from analytical curves.

Apparatus

3. (*a*) *Excitation Source.*—A 2200-v noncondensed a-c arc source having a controllable current output up to 6 amp. The current selected shall be maintained constant to ±0.1 amp.

(*b*) *Spectrograph.*—A spectrograph having a reciprocal linear dispersion of approximately 5 A per mm at 3200 A. In order to record the strontium line at 4077.71 A at a desirable density level a three-step neutral filter, transmitting approximately 5, 20, and 100 per cent of the incident light, shall be placed at the stigmatic focal point of the spectrograph.

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[1] This suggested method has no official status in the Society but is published as information only. The method is based on the experience of the submitters. Comments are solicited.

This method is a revision of Suggested Method E-2 SM 10-8, which was published in "Methods for Emission Spectrochemical Analysis," Am. Soc. Testing Mats. (1957).

[2] This suggested method was prepared by the following members of Task Group 1 of E-2 Subcommittee X on Glass, Ceramics, Alkalies, and Cementitious Materials:

W. H. Koch, Olin-Mathieson Chemical Corp., Niagara Falls, N. Y.

G. Oplinger, Solvay Process Division, Allied Chemical & Dye Corp., Syracuse, N. Y.

[3] O. S. Duffendack and R. A. Wolfe, "Analysis of Caustic Liquors for Traces of Impurities," *Industrial and Engineering Chemistry*, Analytical Edition, Vol. 10, pp. 161–164 (1938).

(*c*) *Developing Equipment.*—Developing equipment that provides temperature control within ±1 F and continual agitation of the solutions. A plate washer and blower-heater drier shall also be available.

(*d*) *Microphotometer.*—A microphotometer capable of measuring the transmittance of the spectral lines.

(*e*) *Calculating Equipment.*—A calculating board for converting transmittance measurements directly into log intensity ratios or concentration.

TABLE I.—STANDARD METAL STOCK SOLUTIONS.

Element	Concentration, g per ml	Salt	Weight of Salt, g
Silicon	0.04	$Na_2SiO_3 \cdot 9H_2O$	40.5
Strontium	0.02	$Sr(NO_3)_2$	4.83
Calcium	0.02	$Ca(NO_3)_2 \cdot 4H_2O$	11.8
Aluminum	0.02	$Al(NO_3)_3 \cdot 9H_2O$	27.8
Iron	0.01	$Fe(NO_3)_3 \cdot 9H_2O$	7.25
Nickel	0.01	$Ni(NO_3)_2 \cdot 6H_2O$	4.95
Chromium	0.005	$K_2Cr_2O_7$	1.41
Lead	0.005	$Pb(NO_3)_2$	0.80
Vanadium	0.005	$VOSO_4 \cdot 2H_2O$	1.78
Boron	0.005	$Na_2B_4O_7 \cdot 10H_2O$	4.41
Cobalt	0.005	$CoCl_2 \cdot 6H_2O$	2.02
Magnesium	0.005	$Mg(NO_3)_2 \cdot 6H_2O$	5.28
Copper	0.005	$Cu(NO_3)_2 \cdot 3H_2O$	1.9
Manganese	0.005	$MnCl_2 \cdot 4H_2O$	1.8

Purity and Concentration of Reagents

4. The purity and concentration of chemical reagents shall conform to the requirements prescribed in the Recommended Practices for Apparatus and Reagents for Chemical Analysis of Metals (ASTM Designation: E 50).[4]

Reagents and Materials

5. (*a*) *Electrodes.*—High-purity graphite rods $\frac{1}{4}$ in. in diameter and $\frac{1}{2}$ in. in length shall be used. One end of each electrode shall be provided with a slightly bevelled edge and the surface shall be treated by polishing on filter paper, or in any suitable manner, to prevent undue absorption of the sample.

(*b*) *Photographic Emulsion.*—Eastman Spectrum Analysis No. 2, Eastman No. 33, or equivalent shall be used.

(*c*) *Photographic Processing Solutions.*—The formulas for photographic processing solutions are given in the Recommended Practices for Photographic Processing in Spectrochemical Analysis (ASTM Designation: E 115).[5]

(*d*) *Standard Caustic Soda Solution (1 ml = 0.64 g NaOH).*—Prepare spectrographically pure sodium hydroxide (NaOH) by repeated electrolysis of high-purity caustic soda in a stationary mercury cell made of methyl methacrylate. Alternatively, the purest reagent-grade NaOH may be used. Adjust the concentration to 640 g NaOH per liter and store in a polyethylene container.

(*e*) *Standard Metal Stock Solutions.*—Prepare standard stock solutions of the metals, as indicated in Table I. Dissolve in water and dilute to 100 ml in a volumetric flask.

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[4] "Methods for Emission Spectrochemical Analysis," Am. Soc. Testing Mats., p. 41 (1957).

[5] "Methods for Emission Spectrochemical Analysis," Am. Soc. Testing Mats.. p. 1 (1957).

(*f*) *Standard Molybdenum Solution* (*1 ml = 0.0004 g Mo*) (Note 1).—Dissolve 12 g of sodium molybdate ($Na_2MoO_4 \cdot 2H_2O$) in water and dilute to 1 liter to make a solution slightly stronger than required. Standardize according to the procedure described in the Recommended Practices for Apparatus and Reagents for Chemical Analysis of Metals (ASTM Designation: E 50).[6] Dilute the solution to the required concentration.

Note 1.—The concentration of molybdenum may need to be modified, depending on the excitation and spectrograph used. The quantity for a given procedure must be constant and standardized.

TABLE II.—ALIQUOT VOLUMES FOR STANDARDS.

Standard	Standard Caustic Soda Solution, ml	Standard Molybdenum Solution, ml	Standard Working Impurity Solution, ml	Water, ml
1 (blank)	25	10	0	15
2	25	10	0.20	14.8
3	25	10	0.80	14.2
4	25	10	2.0	13
5	25	10	5.0	10
6	25	10	14.0	1

(*g*) *Standard Working Impurity Solution* (*element concentration = 0.01 of Table I concentration*).—Combine 1.0-ml aliquots of each of the individual metal stock solutions (Paragraph (*e*)) in a 100-ml volumetric flask, as follows. First add 1.0-ml aliquots of all the metal solutions except the silicon and boron solutions. Then dilute to about 50 ml with water, add 2 ml of concentrated nitric acid (HNO_3, sp gr 1.42) and mix well. Now add the 1.0-ml aliquots of silicon and boron slowly while swirling the solution. Dilute with water to 100 ml and mix well. Prepare fresh for use.

Preparation of Standards

6. (*a*) Synthesized standards, prepared by adding known quantities of impurity solution (Section 5 (*g*)) to pure caustic soda solutions, shall be used. Prepare the standards shown in Table II in polyethylene bottles, mixing them well (Note 2).

Note 2.—Alkaline solutions stored in polyethylene bottles are not stable in respect to the contained metallic impurities, which "plate out" on the inner walls. Standards and samples should be analyzed soon after preparation. Polyethylene bottles thus contaminated can be cleaned by rinsing with HNO_3 (1:1) followed by distilled water.

(*b*) Table III gives the weights of added elements present in 50-ml volumes of standards prepared in accordance with Paragraph (*a*) and the concentration of the elements in the standards.

Preparation of Sample

7. (*a*) If the sample is anhydrous caustic soda, weigh 16.0 g in a polyethylene beaker, dissolve in a minimum of water, add 10.0 ml of molybdenum solution (1 ml = 0.0004 g Mo), and mix well. Transfer to a polyethylene bottle, dilute to 50 ml with water, and mix well (Note 2).

(*b*) For 50 per cent caustic soda liquors, weigh a 32.0-g sample and prepare as in Paragraph (*a*).

(*c*) For other concentrations, determine the sodium hydroxide (NaOH) content and use a weight of sample equivalent to 16.0 g NaOH. Prepare as in Paragraph (*a*).

Electrode System

8. (*a*) Place pairs of the prepared electrodes (Section 5 (*a*)) in a suitable tray or plate (stainless steel, nickel, etc.). On the polished end of each of the pair of electrodes, place a drop of the prepared sample solution. Prepare the pairs in triplicate. Dry the electrodes in an

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[6] 1956 Book of ASTM Methods for Chemical Analysis of Metals, p. 31.

TABLE III.—ELEMENT WEIGHTS AND CONCENTRATIONS IN STANDARDS.[a]

Element	Standard No. 1 (blank)[b]		Standard No. 2		Standard No. 3		Standard No. 4		Standard No. 5		Standard No. 6	
	Weight, g	Concentration, ppm	Weight, g	Concentration, ppm	Weight, g	Concentration, ppm	Weight, g	Concentration, ppm	Weight, g	Concentration, ppm	Weight, g	Concentration, ppm
Silicon	0	0	0.00008	5	0.00032	20	0.0008	50	0.0020	125	0.0056	350
Strontium Calcium Aluminum	0	0	0.00004	2.5	0.00016	10	0.0004	25	0.0010	62.5	0.0028	175
Iron Nickel	0	0	0.00002	1.3	0.00008	5	0.0002	12.5	0.0005	31	0.0014	88
Chromium Lead Vanadium Boron Cobalt Magnesium Copper Manganese	0	0	0.00001	0.6	0.00004	2.5	0.0001	6.3	0.00025	15.6	0.0007	44

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[a] The values given for concentration are based on anhydrous caustic soda.

[b] Residual impurity content is evaluated according to Section 12(*b*).

atmosphere of carbon dioxide, either in an oven or over a micro burner.

(*b*) Place the electrode pair vertically in the arc stand so that the ends bearing the dried sample are nearly in contact. During the preburn adjust the position of the arc on the optical axis of the spectrograph. Set the analytical gap width to 2 mm.

NOTE 3.—In order to record the strontium line at 4077.71 A at a desirable density, a 3-step neutral filter, transmitting 5, 20, and 100 per cent of the incident light, is placed at the stigmatic focal point of the spectrograph. Alternatively, a blue gelatin filter having a transmission of 8 per cent at 4000 A is placed over the 3800 to 4100 A region, while a neutral filter having a 50 per cent transmission is placed directly over the 4077.71 A line.

TABLE IV.—ANALYTICAL LINE PAIRS.

Element	Analytical Line, A	Internal Standard Line, A	Concentration Range, ppm
Silicon	Si 2881.58	Mo 3112.12	4 to 300
Strontium	Sr 4077.71	Mo 3902.96	4 to 150
Calcium	Ca 3968.47	Mo 3902.96	2 to 150
Aluminum	Al 3092.71	Mo 3112.12	0.8 to 150
Iron	Fe 3021.07	Mo 3112.12	4 to 75
Nickel	Ni 3414.77	Mo 3405.94	1.5 to 75
Chromium	Cr 2835.64	Mo 2816.15	4 to 40
Lead	Pb 2833.07	Mo 2816.15 or Mo 3112.12	4 to 40
Vanadium	V 3184.00	Mo 3112.12	2 to 40
Boron	B 2497.33 or 2496.78	Mo 2644.35	0.4 to 40
Cobalt	Co 3453.51	Mo 3112.12	0.4 to 40
Magnesium	Mg 2802.70	Mo 2816.15	0.4 to 40
Copper	Cu 3247.54	Mo 3112.12	0.1 to 40
Manganese	Mn 2794.82	Mo 3112.12	0.1 to 40

Excitation and Exposure

9. Produce and record the spectrum according to the following conditions:

(*a*) *Electrical Parameters:*

Secondary voltage, v	2200
Current, amp	3 to 6

The electrical parameters should be measured as described in the Suggested Practices for Describing and Specifying the Excitation Source (E-2 SM 1-2).[7]

(*b*) *Exposure Conditions:*

Spectral region, A	2400 to 4100
Slit width, μ	40 to 50
Filter	as required (Note 3)
Preburn, sec	30
Exposure period	as required (Note 4)

NOTE 4.—Specific exposure conditions must be determined for each installation. A 90-sec exposure normally is adequate, except for boron which may require exposures up to 5 min.

(*c*) *Replicate Exposures.*—Make triplicate exposures of standards and samples. Once analytical curves are established, it is recommended that exposures of at least two standards be made on each plate to check for curve drift.

(*d*) *Calibration Exposure.*—Produce a spectrum of pure iron or carbon steel, using the same excitation conditions as for the sample. Reduce arc intensity by means of suitable neutral screens.

Photographic Processing

10. Process the emulsion in accordance with the Recommended Practices for

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[7] "Methods for Emission Spectrochemical Analysis," Am. Soc. Testing Mats., p. 68 (1957).

Photographic Processing in Spectrochemical Analysis (ASTM Designation: E 115).[5]

Photometry

11. Measure the transmittance of the spectrum line pairs listed in Table IV for the standards and samples.

Calibration

12. (*a*) *Emulsion Calibration.*—Construct an emulsion calibration curve from the calibration spectra transmittances, following the directions described in the Recommended Practices for Photographic Photometry in Spectrochemical Analysis (ASTM Designation: E 116).[8]

TABLE V.—PRECISION OF METHOD.

Element	Average Concentration, ppm	Coefficient of Variation[a]	Number of Determinations
Si	93	5.7	8
Ca	34	5.6	8
Al	8.5	8.8	8
Fe	15	6.1	8
Mg	15	8.4	8
Cu	1	0	8

[a] Coefficient of variation, ν, in this method, is calculated as follows:

$$\nu = \frac{100}{\bar{X}} \sqrt{\frac{\Sigma d^2}{n-1}}$$

where:

$\bar{X}$ = average concentration, in ppm,
d = difference of the determination from the mean, and
n = number of determinations.

(*b*) *Preparation of Analytical Curves.*—Convert the transmittances of the analytical and internal standard lines into log intensity ratios, using the emulsion calibration curve prepared in Paragraph (*a*) or by means of a calculating board. For each element prepare an analytical curve relating the log intensity ratios to concentration. Transcribe appropriate scales for use in the calculating board, if desired. Make correction for residual impurity in the blank by the method of successive approximations[9] or by the zero intercept method.[10]

Calculations

13. (*a*) Convert the transmittance measurements of the line pairs to log intensity ratios by means of the emulsion calibration curve or by means of the calculating board. Determine concentration from the analytical curves. If analytical scales have been prepared, transmittance measurements can be reduced to concentration directly by means of the calculating board. Compensate for analytical curve drift by applying a correction as determined from the observed value of the standard samples.

(*b*) The concentrations, as determined from the analytical curves, are based on anhydrous caustic soda. For anhydrous caustic soda samples the concentrations of the impurities determined apply directly. For caustic soda liquor samples the concentration of the impurities read from the analytical curves must be multiplied by the percentage of NaOH in the sample to give the concentration of the impurities on the sample basis.

Precision and Accuracy

14. (*a*) *Precision.*—Data in Table V indicate the precision of the method realized for some of the elements in one laboratory.

(*b*) *Accuracy.*—Experience in several laboratories indicates the accuracy approximates ±10 per cent of the amount present, based on comparisons with colorimetric analysis.

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[8] "Methods For Emission Spectrochemical Analysis," Am. Soc. Testing Mats., p. 12 (1957).

[9] W. C. Pierce and N. H. Nachtrieb, "Photometry in Spectrochemical Analysis," *Industrial and Engineering Chemistry*, Analytical Edition, Vol. 13, pp. 774–781 (1941).

[10] Jacob Cholak and Robert V. Story, "Corrections for Residual Impurity and Background in Spectrochemical Analysis," *Journal*, Optical Soc. America, Vol. 32, pp. 502–505 (1942).

REPORT OF COMMITTEE E-3

ON

CHEMICAL ANALYSIS OF METALS*

Committee E-3 on Chemical Analysis of Metals held one meeting during the year: on June 19, 1957, in Atlantic City, N. J., in conjunction with the Annual Meeting of the Society. Divisional and other task groups held individual sessions on June 16 to 19, 1957.

The consideration and development of newer methods of analysis continues as a dynamic process within the committee and it is keeping pace with the newer concepts in alloy and material development. Several representatives of Committee E-3 were present at the First Annual Analytical Chemical Symposium on Nuclear Materials. At present one task group is concerned with nuclear grade zirconium. Other refractory alloys are under consideration.

A successful Symposium on the Determination of Gases in Metals was sponsored by the committee on June 18, 1957 at the Annual Meeting of the Society and has been issued as ASTM STP 222. Initial communications have been made to integrate with the British Standards Institution and the British Iron and Steel Research Assn. on common problems. An early exchange of information is contemplated.

NEW TENTATIVES

The committee recommends for publication as tentative the following methods as appended hereto:[1]

Method for:

Determination of Arsenic in Fire-Refined Copper, and

Chemical Analysis of Magnesium and Magnesium-Base Alloys.

Supporting data for these two proposed new tentatives are given in the accompanying Tables I and II.

ADOPTION OF TENTATIVES AS STANDARD WITHOUT REVISION

The committee recommends that the following tentative methods be approved for reference to letter ballot of the Society for adoption as standard without revision:

Tentative Methods for Chemical Analysis of:

Ferro-Alloys (**E 31 – 54 T**),[2]

Nickel-Chromium and Nickel-Chromium-Iron Alloys (**E 38 – 56 T**),[2]

Slab Zinc (Spelter) (**E 40 – 56 T**),[2]

Zinc-Base Die-Casting Alloys (**E 47 – 56 T**),[2]

Sulfur in Special Brasses and Bronzes (**E 54 – 50 T**),[2]

Copper-Beryllium Alloys (**E 106 – 54 T**),[2]

Tentative Photometric Methods for Chemical Analysis of:

Aluminum-Base Alloys (**E 34 – 56 T**),[2]

Lead, Tin, Antimony and Their Alloys (**E 87 – 56 T**),[2] and

Tentative Methods of Sampling:

Non-Ferrous Metals and Alloys in Cast Form for Determination of Chemical Composition (**E 88 – 50 T**).[2]

REAPPROVAL OF STANDARDS

The committee recommends the re-

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* Sixty-first Annual Meeting of the Society, June 22–27, 1958.

[1] The new tentatives are available as separate reprints.

[2] 1956 Book of ASTM Standards on Chemical Analysis of Metals.

TABLE I.—SUPPORTING DATA FOR METHOD FOR DETERMINATION OF ARSENIC IN FIRE-REFINED COPPER BY BUTANOL EXTRACTION.

Arsenic as determined by distillation and titration using 50 g sample, 0.0054 per cent.

	Laboratory A	Laboratory B	Laboratory C	Laboratory D	Laboratory E
Minimum	0.0057	0.0057	0.0056	0.0056	0.0058
Maximum	0.0061	0.0063	0.0057	0.0060	0.0061
Average	0.0059	0.0059	0.0057	0.0058	0.0059

TABLE II.—SUPPORTING DATA FOR PROPOSED TENTATIVE METHOD OF CHEMICAL ANALYSIS OF MAGNESIUM AND MAGNESIUM-BASE ALLOYS.

Zirconium by the Alizarin Red-S (Photometric) Method.

	Zirconium, per cent		
	Soluble	Insoluble	Total
Sample No. 82411A-EK 30:			
Laboratory A	0.27	0.008	0.28
	0.28	0.008	0.29
Laboratory B	0.25	0.015	0.27
	0.27	0.022	0.29
Laboratory C	0.27	0.009	0.28
	0.26	0.007	0.27
Laboratory D	0.26	0.004	0.26
	0.29	0.007	0.30
	0.28	0.010	0.29
Average	0.27	0.010	0.28
Sample No. 82507A-HK 31:			
Laboratory A	0.58	0.014	0.59
	0.58	0.014	0.59
Laboratory B	0.58	0.022	0.60
	0.58	0.022	0.60
Laboratory C	0.61	0.016	0.63
	0.60	0.014	0.61
Laboratory D	0.58	0.007	0.59
	0.62	0.012	0.64
	0.58	0.016	0.60
Average	0.59	0.015	0.61

Gravimetric Determination of Thorium.

	Thorium, per cent	
	Sample No. 82507A	Sample No. 83190A
Laboratory A	3.13	1.15
	3.12	
Laboratory B	3.08	1.16
	3.12	1.17
	3.11	1.21
	3.08	1.23
Laboratory C	3.17	1.26
	3.12	1.24
	3.14	1.25
		1.24
Average	3.12	1.21

Gravimetric Determination of Rare Earth Elements.

	Rare Earths, per cent	
	Sample No. 82981A	Sample No. 82411A
Laboratory A	1.43	3.70
	1.40	3.77
Laboratory B	1.44	3.60
	1.42	3.71
	1.41	3.68
	1.41	3.73
Laboratory C	1.45	3.70
	1.43	3.73
	1.45	3.74
Average	1.43	3.71

approval of the following standards which have stood for more than six years without revision:

Standard Methods of:

Sampling Ferro-Alloys (**E 32 – 42**),

Chemical Analysis of Copper (Electrolytic Determination) (**E 53 – 48**),

Sampling Wrought Non-Ferrous Metals and Alloys for Determination of Chemical Composition (**E 55 – 48**), and

Standard Methods for:

Chemical Analysis of Brasses (**E 36 - 45**).

TENTATIVES CONTINUED WITHOUT REVISION

The committee recommends the continuation without revision of the following tentatives:

Tentative Methods for Chemical Analysis of:

Electronic Nickel (**E 107 - 56 T**),
Copper-Chromium Alloys (**E 118 - 56 T**),
Titanium and Titanium Base Alloys (**E 120 - 56 T**),
Copper-Tellurium Alloys (**E 121 - 56 T**),

Tentative Photometric Methods for Determination of:

Phosphorus in High Phosphorus Brazing Alloys (**E 56 - 56 T**), and

Tentative Recommended Practices for Photometric Methods for:

Chemical Analysis of Metals (**E 60 - 50 T**).

STANDARD CONTINUED WITHOUT REVISION

The committee recommends the continuation without revision of the Standard Methods for Chemical Analysis of Silver Solders (**E 56 - 45**), subject to study of the validity of the procedure in part.

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[3]

ACTIVITIES OF SUBCOMMITTEES

Editorial Subcommittee (J. L. Hague, chairman).—Agreement in principle was reached on the format of manual to cover the by-laws and editorial policies of the committee. The new photometric method for cobalt in stainless steels was edited in detail by the committee as an example of styling.

Division S on Sampling (A. C. Holler, chairman):

Subcommittee S-1 on Ferrous Alloys (J. B. Armstrong, chairman) is rewriting the sampling method for ferro alloys.

Subcommittee S-2 on Non-Ferrous Alloys (R. L. Vitek, chairman).—Methods for sampling titanium sponge are being prepared jointly with Subcommittee VIII on Miscellaneous Refined Metals and Alloys of Committee B-2.

Division G on General Analytical Methods (H. F. Beeghly, chairman):

A series of symposia is being sponsored which are intended to stimulate development of improved methods for analysis of metals and the evolution of common analytical procedures.

A Symposium on Gases in Metals was sponsored at the 1957 Annual Meeting of the Society and has been issued as ASTM STP 222. A Symposium on Solvent Extraction in Metals Analysis is being sponsored at the 1958 Annual Meeting.[4] The 1956 Symposium on Ion Exchange and Chromatography in Analytical Chemistry is available as ASTM STP 195.

The methods prepared by Committee E-3 have been reviewed with the object of determining when modifications can be introduced to provide more uniform procedural detail in the next edition of the book "ASTM Methods for the Analysis of Metals."

Division M on Miscellaneous Metals (R. M. Fowler, chairman):

A task group has been organized for the analysis of zirconium and its alloys. It plans to have preliminary drafts of several methods available by the time of the Annual Meeting.

Methods for powder metals are being

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[3] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

[4] To be issued as separate publication *ASTM STP No. 238.*

developed by two separate task groups. Methods for chemical analysis, nonvolatile material in tungsten powder, and hydrogen loss of metal powders are under development.

A task group to consider the methods for the analysis of columbium has been initiated.

Division F on Ferrous Metals (J. J. Furey, chairman):

Task groups are at work on the following projects:

Photometric Method for Aluminum in Stainless Steel

The Aluminon Method has been set aside, and two new methods are being studied. Samples have been distributed.

Hydrogen in Steel

Samples were distributed and statistical study of preliminary results has been made. The variability is such that more work must be done, and another meeting of the task group has been scheduled to determine types of samples to be used on the next test.

Cobalt (Titrimetric) in High-Alloy Materials

Three methods have been evaluated and in each case one or more laboratories encountered trouble. Further testing of another method submitted is under way.

Copper in Steel

Results indicate that the method is accurate and reproducible in the range of 0.017 per cent to 1.5 per cent copper.

Nitrogen in Steel

This project contemplates determination of both soluble and insoluble nitrogen. A method has been agreed upon and samples will be distributed consisting of a plain carbon steel with low nitrogen content, a stainless steel, and a high-temperature alloy with 0.15 per cent to 0.20 per cent nitrogen.

Cobalt (Photometric) in Stainless Steel

A very satisfactory method had been developed. Further work on an alternate method is still under way.

Phosphorus (Photometric) in Steel

Preliminary data indicate that a method under study will give reliable results in the range of 0.008 per cent to 0.88 per cent phosphorus. A tungsten steel and a titanium-bearing steel will be included in the evaluation program.

Ferroboron - Ferrocolumbium

Boron results by the ion-exchange method appear quite satisfactory. A photometric method for aluminum is now being evaluated, and it is hoped that this will eliminate the difficulty experienced with the gravimetric procedure. Committee A-9 is writing specifications for ferrocolumbium. It is planned to set up a standard sample with the National Bureau of Standards in connection with the development of ASTM methods.

New task groups have been appointed to investigate a method for carbon in the triple 0 range, a method for sulfur in the range of 0.0005 per cent to 0.001 per cent, and a task group will be set up to study the Lingane method for manganese as a substitute or alternate method for determination of manganese in ferromanganese and silicomanganese.

Division N on Non-Ferrous Metals (R. G. Ernst, chairman) prepared the proposed new tentative methods referred to earlier in the report.

Methods for the photometric determination of magnesium and the gravimetric determination of tungsten in electronic nickel have been received from the Committee F-1 task force, and are being prepared for inclusion in the Methods for Chemical Analysis of Electronic Nickel (E 107).

Methods being evaluated include the photometric determination of antimony in pig lead and fire-refined copper with rhodamine-B, the application of neo-cuproine to the determination of small amounts of copper in various materials, the determination of zinc in aluminum alloys by thiocyanate extraction and EDTA titration, the determination of cadmium in silver solders by carbamate extraction and EDTA titration, and the photometric determination of bismuth in fire-refined copper by the iodide method. It is hoped that cooperative data on these methods will be available in the near future.

Work is continuing on methods for the determination of selenium and tellurium in fire-refined copper and the determination of trace amounts of aluminum in slab zinc. A new task group was activated to study the determination of small amounts of boron and beryllium in aluminum-base alloys. It is planned to review all existing methods in the near future with the intent of modernizing older procedures when possible and taking full advantage of the latest developments in analytical chemistry.

This report has been submitted to letter ballot of the committee, which consists of 129 members; 65 members returned their ballots, of whom 64 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

ARBA THOMAS,
Chairman.

H. KIRTCHIK,
Secretary.

REPORT OF COMMITTEE E-4
ON
METALLOGRAPHY*

Committee E-4 on Metallography, together with several of its subcommittees, held two meetings during the the past year: in Atlantic City, N. J., on June 17 and 18, 1957, and in St. Louis, Mo., on February 10 and 11, 1958.

The committee now consists of 110 members.

The officers elected for the ensuing term of two years are as follows:

Chairman, L. L. Wyman.
Vice-Chairman, R. E. Penrod.
Secretary, Mary R. Norton.

REVISIONS OF TENTATIVES

The committee recommends revisions as indicated of the following tentatives and their continuation as tentative:

Methods of Preparation of Metallographic Specimens (E 3 – 46 T):[1]

Revise by the deletion of Sections 7 to 11, replacing them with new material on electrolytic polishing, as appended hereto.[2]

Methods of Determining the Average Grain Size of Metals (E 112 – 55 T):[3]

Revise as appended hereto.[2]

STANDARD CONTINUED WITHOUT REVISION

The committee recommends the continuation without revision of the Standard Recommended Practice for Determining the Inclusion Content of Steel (**E 45 – 51**).

TENTATIVES CONTINUED WITHOUT REVISION

The committee recommends the continuation without revision of the following tentatives:

Tentative Methods of:

Preparation of Micrographs of Metals and Alloys (Including Recommended Practice for Photography as Applied to Metallography (**E 2 – 49 T**),
Preparing Quantitative Pole Figures of Metals (**E 81 – 54 T**), and
Determining the Orientation of a Metal Crystal (**E 82 – 49 T**).

Tentative Definitions of:

Terms Relating to Metallography (**E 7 – 55 T**).

Tentative Recommended Practice for:

Thermal Analysis of Metals and Alloys (**E 14 – 57 T**), and
Dilatometric Analysis of Metallic Materials (**E 80 – 49 T**).

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Selection and Preparation of Samples (F. C. Hull, chairman) has completed a new section on electrolytic polishing to replace the present version in Methods E 3 referred to earlier in the report.

Subcommittee III on Nomenclature (P. A. Beck, chairman) has collaborated with Society Headquarters in having the subcommittee proposal of a system of phase nomenclature distributed to the technical organizations both here and abroad in order to elicit suggestions.

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* Sixty-first Annual Meeting of the Society, June 22–27, 1958.

[1] 1955 Book of ASTM Standards, Part 2.

[2] The revised tentatives appear in the 1958 Book of ASTM Standards, Part 3.

[3] 1956 Supplement to Book of ASTM Standards, Part 1.

Subcommittee V on Micro Hardness (W. E. Shebest, chairman) is continuing its activity in round-robin tests using specially calibrated reference standards.

Subcommittee VI on X-ray Methods (W. L. Fink, chairman) has been engaged in writing completely new recommended practices for powder diffraction work. The several task groups engaged in the various phases of this work are nearing the ends of their respective assignments.

Subcommittee VII on Thermal Analysis (D. I. Finch, chairman) has drawn up an initial draft for a recommended practice for temperature *versus* electrical resistance methods.

Subcommittee VIII on Grain Size (R. E. Penrod, chairman) has completely revised the numerical tables in Methods E 112 to give them the highest possible accuracy; it has added sections on sample treatment and on the reporting of austenitic grain sizes which were particularly requested by steel industry representatives; it has selected a suitable material for the preparation of Plate II, twinned-grains, flat etch, and is preparing the standards.

Additional grain size micrographs have been prepared for the inclusion of new sizes in Plate III, as requested by Committee B-5 on Copper and Copper Alloys. Also, cooperative effort with Committee B-2 on Non-Ferrous Metals and Alloys has resulted in the use of Methods E 112 as the grain-size method for six nickel-base alloy specifications.

Subcommittee XI on Electron Microstructure of Metals (S. T. Ross, chairman) has extended its activities into the nonferrous fields, with several active subgroups being concerned with special interest areas as, for example, the precipitates in some super strength alloys.

The subcommittee sponsored the Symposium on Advances in Electron Metallography at the 1958 Annual Meeting.[4] In addition, the subcommittee is holding a special meeting for those interested and potentially active in electron probe (micro spot X-ray analyzer) work, preparatory to organizing a subgroup in this new field of effort.

This report has been submitted to letter ballot of the committee, which consists of 110 members; 91 members returned their ballots, of whom 87 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

L. L. WYMAN,
Chairman.

MARY R. NORTON,
Secretary.

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[4] Issued as a separate publication *ASTM STP No. 245.*

REPORT OF COMMITTEE E-5
ON
FIRE TESTS OF MATERIALS AND CONSTRUCTION*

Committee E-5 on Fire Tests of Materials and Constructions held two meetings since its last report in 1956: in Philadelphia, Pa., on February 8, 1957, and in St. Louis, Mo., on February 19, 1958.

The committee consists of 62 members of whom 29 are classified as producers, 6 as consumers, and 27 as general interest, with 3 consulting members.

The officers elected for the ensuing term of two years are as follows:

Chairman, W. J. Krefeld.
Vice-Chairman, R. C. Corson.
Secretary, H. D. Foster.

New Tentative

The committee recommends for publication as tentative the Method of Test for Defining Noncombustibility of Building Materials as appended hereto.[1]

Adoption of Tentative Revisions as Standard

The committee recommends that the tentative revisions[2] of Section 26, issued June, 1956, of Standard Methods of Fire Tests of Building Construction and Materials (**E 119 – 55**)[3] be approved for reference to letter ballot of the Society for adoption as standard.

Revision of Standard, Immediate Adoption

The committee recommends for immediate adoption revisions as follows of the Standard Methods of Fire Tests of Building Construction and Materials (**E 119 – 55**)[3] and accordingly asks for the necessary nine-tenths affirmative vote at the Annual Meeting in order that this revision may be referred to letter ballot of the Society:

Section 5.—Reletter Paragraph (*b*) as (*c*) and add a new Paragraph (*b*) as follows:

> (*b*) Reports of tests involving wall, floor, beam or ceiling constructions in which restraint is provided against expansion, contraction and/or rotation of the construction shall describe the method used to provide this restraint.

Section 23.—Change the heading "Size of Sample" to read "Size and Construction of Sample." Add the following as Paragraph (*b*):

> (*b*) Beams or joists forming part of the assembly shall be supported in accordance with the recommended fabrication procedures for the type of construction. Assemblies representing forms of construction which restrain structural elements and top deck shall be supported by a restraining frame incorporated in the furnace structure simulating such restraint.

Adoption of Tentatives as Standard

The committee recommends that the following tentatives be approved for reference to letter ballot of the Society for adoption as standard:

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* Sixty-first Annual Meeting of the Society, June 22–27, 1958.

[1] The new tentative appears in the 1958 Book of ASTM Standards, Part 5.

[2] 1956 Supplement to Book of ASTM Standards, Part 4.

[3] 1955 Book of ASTM Standards, Part 4.

Tentative Methods of:

Fire Tests of Door Assemblies (**E 152 - 56 T**),[3] and

Fire Tests of Roof Coverings (**E 108 - 56 T**).[3]

REAPPROVAL OF STANDARDS

The committee recommends the reapproval of the following standards which have stood for more than six years without revision:

Standard Method of Test for:

Combustible Properties of Treated Wood by the Fire-Tube Apparatus (**E 69 - 50**), and

Combustible Properties of Treated Wood by the Crib Test (**E 160 - 50**).

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[4]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Fire Tests of Materials and Constructions (A. J. Steiner, chairman).—Additional improvements of Methods E 119 which are being considered include (a) controlled conditioning of fire test specimens, (b) a more exact method of determining time of load failure of floors under fire exposure, (c) a better correlation of those sections dealing with fire tests of floors and roofs and of fire tests of ceiling constructions, (d) the need for long-span furnaces for testing beams, (e) the need of roof coverings on test specimens for fire tests of roof construction, (f) the use of the word "combustible" with fire test ratings where applicable, and (g) the development of a suitable surface thermocouple protection pad to replace the one described in Section 4(*a*), which is unobtainable.

Subcommittee III on Fire Tests of Door Assemblies (H. D. Foster, chairman).—The need for methods for fire tests of windows is being considered.

Subcommittee IV on Fire Tests of Acoustical and Similar Finishes (C. H. Yuill, chairman).—The Tentative Method E 84 - 50 T was editorially revised and an illustration showing the basic dimensions of the test furnace was provided. Other methods and equipment, particularly that developed by the Forest Products Laboratories, are being considered for use in determining the flame-spread ratings of interior finishes.

Subcommittee V on Nomenclature and Definitions (J. R. Shank, chairman) prepared the Test Method for Defining Noncombustibility of Building Materials which is referred to earlier in the report. Definitions for other terms used in fire testing are being considered.

Subcommittee VIII on Fire Tests of Roof Coverings (E. W. Fowler, chairman). —The Method of Fire Tests of Roof Coverings (E 108 - 56 T) has been recommended for adoption as standard. The classifications of roof coverings used by the Underwriters' Laboratories, Inc. and in most building codes are determined by tests made in conformity with these methods.

Subcommittee X on Research (A. F. Robertson, chairman).—This is a new subcommittee organized to encourage and promote research in the fire protection field.

This report has been submitted to letter ballot of the committee, which consists of 62 voting members; 60 members returned their ballots, of whom 53 have voted affirmatively, and 0 negatively.

Respectfully submitted on behalf of the committee,

W. J. KREFELD,
Chairman.

H. D. FOSTER,
Secretary.

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[4] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

REPORT OF COMMITTEE E-6

ON

METHODS OF TESTING BUILDING CONSTRUCTIONS*

Committee E-6 on Methods of Testing Building Constructions and its subcommittees held one meeting during the year: on October 9 to 11, 1957, in Ottawa, Canada, in conjunction with meetings of Committees C-16 on Thermal Insulating Materials and C-20 on Acoustical Materials.

The committee is sponsoring a Symposium on Durability at the 1958 Annual Meeting of the Society.[1]

The committee consists of 96 members, of whom 75 are voting members; 32 are classified as producers, 8 as consumers, and 35 as general interest members. There are 2 consulting members.

The officers elected for the ensuing term of two years are as follows:

Chairman, R. F. Legget.
Vice-Chairman, Harold Perrine.
Secretary, R. A. Biggs.
Membership Secretary, J. P. Thompson.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee II on Durability (E. V. Shuman, chairman) arranged for the Symposium on Durability referred to earlier in the report.

Subcommittee IV on Strength of Masonry Units (H. C. Plummer, chairman) is progressing on the development of test methods for masonry units.

Subcommittee V on Light Trusses (D. E. Kennedy, chairman), and

Subcommittee VI on Load Tests on Completed Structures (W. R. Schriever):

These subcommittees were reorganized during the year and anticipate the development of loading tests in the near future.

Subcommittee VII on Vapor Barriers Beneath Concrete Slabs on the Ground (R. F. Luxford, chairman) was organized during the year, and plans have been made for the preparation of test methods, using as a guide data prepared by the Forest Products Laboratory and the Building Research Advisory Board.

Subcommittee VIII on Windows (R. B. Crepps, chairman) held a conference to discuss the testing of windows which was attended by representatives of Canadian window manufacturers as well as interested members of the Society. Information will be assembled on the principal phases of window testing which will serve as a basis for the work of the subcommittee.

This report has been submitted to letter ballot of the committee, which consists of 75 voting members; 72 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

R. F. LEGGET,
Chairman.

R. A. BIGGS,
Secretary.

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* Sixty-first Annual Meeting of the Society, June 22–27, 1958.

[1] Issued as separate publication *ASTM STP No. 236.*

REPORT OF COMMITTEE E-7

ON

NONDESTRUCTIVE TESTING*

Committee E-7 on Nondestructive Testing met at Atlantic City, N. J., on June 20, 1957, in conjunction with the Annual Meeting of the Society. Numerous task forces and subcommittees as well as the Executive Council also met at that time. Another series of meetings were held in St. Louis, Mo., in February 1958, during ASTM Committee Week.

The Symposium on Nondestructive Testing, sponsored by Committee E-7 at the Second Pacific Area National Meeting of the Society in 1956, was published in November, 1957, as *ASTM STP No. 213.*

The committee records with regret the death of Noah A. Kahn, former chairman of Subcommittee II of Committee E-7, and long-time active leader and counselor in the work of the committee. Mr. Kahn died suddenly during the 1957 Annual Meeting in Atlantic City. His leadership and wisdom will be sorely missed.

The committee consists of 136 members, plus 21 consulting members.

The officers elected for the ensuing term of two years are as follows:

Chairman, J. H. Bly.
Vice-Chairman, C. H. Hastings.
Secretary, Alexander Gobus.

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1957 Annual Meeting, Committee E-7 presented to the Society through the Administrative Committee on Standards the Tentative Recommended Practice for Fabricating and Checking Aluminum Alloy Ultrasonic Standard Reference Blocks (E 127 - 58 T). This recommendation was accepted by the Standards Committee on April 25, 1958.

NEW TENTATIVE

The committee recommends for publication as tentative the Tentative Method of Wet Magnetic Particle Inspection as appended hereto.[1]

TENTATIVES CONTINUED WITHOUT REVISION

The committee recommends the continuation without revision of the following tentatives:

Industrial Radiographic Terminology for:

Use in Radiographic Inspection of Castings and Weldments (**E 52 - 49 T**).

Recommended Practice for:

Radiographic Testing (**E 94 - 52 T**),
Ultrasonic Testing by the Resonance Method (**E 113 - 55 T**), and
Ultrasonic Testing by the Reflection Method, Using Pulsed Longitudinal Waves Induced by Direct Contact (**E 114 - 55 T**).

Reference Radiographs for:

Inspection of Aluminum and Magnesium Castings (**E 98 - 53 T**), and
Steel Welds (**E 99 - 55 T**).

Reference Photographs for:

Magnetic Particle Indications on Ferrous Castings (**E 125 - 56 T**).

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* Sixty-first Annual Meeting of the Society, June 22–27, 1958.

[1] The new tentative appears in the 1958 Book of ASTM Standards, Part 3.

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[2]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Radiographic Recommended Practice (G. R. Forrer, chairman), has obtained suitable specimens and is preparing radiographs of these specimens, using a wide variety of radiation sources and exposure parameters, as its first step in developing a document or publication correlating the variations in radiographic flaw images with variations in these sources or parameters, especially specimen thickness. It is hoped to have these initial radiographs completed by the time of the Annual Meeting.

Subcommittee II on Reference Radiographs (S. Low, chairman).—Work is continuing on the expansion of the tentative reference radiographs of steel welds. A task group is studying the question of suitability of re-issuance of Reference Radiographs E 71 and E 98 in Keysort card form rather than in album form. The Task Force on Reference Radiographs of Heavy Steel Castings has been extremely active and is engaged in the preparation of thick cast steel plates containing specific flaws which are being radiographed using various high-energy radiations to determine the need for separate sets of reference radiographs for the individual sources. These films are also supplemented by numerous commercial radiographs received through liaison with the cast steel industry. Additional liaison has been established with other national groups interested in this field.

The subcommittee has proposed, and has agreed to take the initiative in exploring, that a suitable memorial for Mr. Noah Kahn, deceased chairman of Subcommittee II, be established by Committee E-7.

Subcommittee III on Magnetic Particle and Penetrant Inspection (H. Migel, chairman) completed the proposed Tentative Method of Wet Magnetic Particle Testing referred to earlier in this report. The Task Force on Liquid Penetrant Testing hopes to have a first draft of a method available for review at the time of the Annual Meeting.

Subcommittee V on Radiographic Procedure (C. H. Hastings, chairman) is preparing a proposed Tentative Method of Determining Quality of Radiographic Testing. This document, although firmly agreed on, will incorporate textual material which is considered preliminary; the subcommittee hopes to obtain comments from users to assist in determining whether further revisions will be in order.

Subcommittee VI on Ultrasonic Testing Procedure (J. C. Smack, chairman) completed the development of the Tentative Recommended Practice for Fabricating and Checking Aluminum Alloy Ultrasonic Standard Reference Blocks referred to above. Task groups are still actively developing recommended practices for ultrasonic inspection of ferrous welds and for ultrasonic testing by the immersion method.

Subcommittee VII on Eddy Current Testing.—During the 1958 Committee Week, the Executive Council agreed on the establishment of this committee. It is planned to hold an organization meeting during the Annual Meeting. Numerous interested members are being invited to attend.

This report has been submitted to letter ballot of the committee, which consists of 136 members; 76 members returned their ballots, of whom 68 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

J. H. BLY,
Chairman.

A. GOBUS,
Secretary.

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[2] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

REPORT OF COMMITTEE E-9

ON

FATIGUE

Committee E-9 held two meetings during the year: on June 17, 1957, at Atlantic City, N. J., and on February 10, 1958, at St. Louis, Mo.

During the year five new members were elected to Committee E-9. The following corresponding members were added: R. Cazaud (France), M. Hempel (Germany), W. Weibull (Sweden), and W. A. Wood (Australia).

Attention is called to the Proceedings of the International Conference on Fatigue (1121 pages) issued by the Institution of Mechanical Engineers (London) in March, 1958. The North American Committee appointed for this extensive project consisted of R. E. Peterson, Chairman, T. J. Dolan, A. M. Freudenthal, O. J. Horger, W. T. Lankford, B. J. Lazan, J. M. Lessells, and J. Marin, all members of Committee E-9.

The officers elected for the ensuing term of two years are as follows:

Chairman, R. E. Peterson.
Vice-Chairman, H. J. Grover.
Secretary, O. J. Horger.
Asst. Secretary, W. T. Lankford.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Research (T. J. Dolan, chairman).—At the June 17 meeting, G. M. Sinclair presented a talk on "The Micromechanism of Fatigue." Dislocations were illustrated by a "bubble raft" movie showing consolidation of vacancies due to cycling. The subcommittee sponsored a Symposium on Basic Mechanisms of Fatigue at the 1958 Annual Meeting in Boston.[1]

Subcommittee II on Papers (D. G. Richards, chairman).—In addition to the Symposium papers, the subcommittee reviewed the papers for two sessions on Fatigue at the 1958 Annual Meeting.

Subcommittee III on Survey (H. J. Grover, chairman).—During the year *ASTM STP No. 9H*, "References on Fatigue—1956," was issued. A survey of current projects is in progress,[2] and a cumulative index is planned.

Subcommittee IV on Large Machines and Test Correlation (J. F. Millan, chairman).—*ASTM STP No. 216*, "Symposium on Large Fatigue Testing Machines and Their Results," has been issued during the year. The symposium, organized by John M. Lessells, was held at the 1957 Annual Meeting.

Subcommittee V on Aircraft Structural Fatigue Problems (P. Kuhn, chairman).—The aircraft structures papers presented at the Second Pacific Coast meeting have been published as *ASTM STP No. 203*, "Fatigue of Aircraft Structures."

Task Force on Statistical Methods (F. B. Stulen, chairman).—The "Tentative Guide for Statistical Analysis of Fatigue Data" has been published as a Supplement to *ASTM STP No. 91*.

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[1] To be issued as separate publication *ASTM STP No. 237*.

[2] "Recent Survey of Current Activities on Fatigue," ASTM BULLETIN, No. 235, Jan., 1959, p. 26.

This report has been submitted to letter ballot of the committee, which consists of 52 members; 27 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

R. E. PETERSON,
Chairman.

O. J. HORGER,
Secretary.

REPORT OF COMMITTEE E-11

ON

QUALITY CONTROL OF MATERIALS*

Committee E-11 on Quality Control of Materials held four meetings since the last report in June 1956: on November 27, 1956 at ASTM Headquarters; on April 29, 1957, at the Carborundum Co., Niagara Falls, N. Y.; and on November 19, 1957, and April 29, 1958 at ASTM Headquarters, Philadelphia, Pa.

Three new members have been appointed to the committee, namely, F. E. Grubbs, Ballistic Research Labs.; J. Mandel, National Bureau of Standards; and W. P. Geopfert, Aluminum Company of America. The committee consists of 24 members.

The officers elected for the ensuing term of two years are as follows:

Chairman, S. Collier.
Vice-Chairman, C. A. Bicking.
Secretary, J. H. Davidson.

H. F. Dodge and O. P. Beckwith were elected as members of the Advisory Committee for a four-year term.

A new Task Group No. 15 on Sampling Plans has been appointed with the assignment of developing a framework of sampling plans for acceptance purposes for ASTM specifications.

The committee is sponsoring a Symposium on Bulk Sampling[1] at the 1958 Annual Meeting of the Society in Boston. B. H. Lloyd is chairman of the Symposium Committee.

REVISION OF STANDARD AND REVERSION TO TENTATIVE

The committee recommends revision, as appended hereto,[2] of the Recommended Practices for Designating Significant Places in Specified Limiting Values (**E 29 - 50**)[3] and its reversion to tentative.

This standard is not intended for application to situations involving addition of a series of numbers but is concerned solely with an individual number and its comparison to some limiting value. The present standard follows American Standard Rules for Rounding Off Numerical Values (ASA No.: Z25.1) and applies to a series of rounded numbers. The revised standard applies solely to an individual number and its comparison with some limiting value.

ADOPTION OF TENTATIVES AS STANDARD WITHOUT REVISION

The committee recommends that the following two tentatives be approved for reference to letter ballot of the Society for adoption as standard without revision:

Tentative Recommended Practice for:

Probability Sampling of Materials (**E 105 - 56 T**),[4] and

Choice of Sample Size to Estimate the Average Quality of a Lot or Process (**E 122 - 56 T**).[4]

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* Sixty-first Annual Meeting of the Society, June 22–27, 1958.

[1] Issued as separate publication *ASTM STP No. 242*.

[2] The revised standard appears in the 1958 Book of ASTM Standards, Part 3.

[3] 1955 Book of ASTM Standards.

[4] 1956 Supplement to Book of ASTM Standards.

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[5]

ACTIVITIES OF SUBCOMMITTEE

Subcommittee I on ASTM Problems (W. R. Pabst, Jr., chairman) sponsored a Round-Table Discussion on Statistical Aspects of ASTM Standards at the 1957 Annual Meeting of the Society in Atlantic City, N. J. The subcommittee has given assistance to technical committees of the Society in the preparation of sampling phases of specifications.

This report has been submitted to letter ballot of the committee which consists of 24 members; 21 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

H. F. DODGE,
Chairman.

O. P. BECKWITH,
Secretary.

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[5] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

REPORT OF COMMITTEE E-12
ON
APPEARANCE*

Committee E-12 on Appearance held two meetings during the year: in Atlantic City, N. J., on June 17, 1957, and in Washington, D. C., on March 24 and 25, 1958.

Two of a total of five research projects proposed by Committee E-12 were accepted by the Inter-Society Color Council for research work. Problem Subcommittees 21 and 22 on Materials Standards (of Appearance), and The Jury System in Evaluating Color Differences, respectively, were organized at the March 24 and 25 meeting of the ISCC (jointly with Committee E-12) at Washington, D. C., with Norman Pugh as chairman of the latter.

In a large measure, the bulk of the work of Committee E-12 is research in nature. The current organization is along lines of parameters of appearance, but these longer term groupings are being reorganized to favor shorter term work groups concerned with the solution of specific problems by personnel particularly suited and selected for such problems.

The more active groups in Subcommittee II on Color and Spectral Characteristics show the value of the latter arrangement. Group 3 on Color Specification of Transparent Colors is working with ISCC's Problem 14 Committee on the Colorimetry of Transparent Materials, and may produce a highly useful as well as original approach to the industry-wide problem of using single-number color ratings for materials of commercial importance such as oils, plastics, varnishes, etc. Group 6 on Color of Petroleum, Coal-Tar and Related Resins may develop an improved instrumental technique for measuring color of these resins.

A special group of Committee E-12 members is progressing in the development of a "Manual for Appearance Evaluation of Materials." This will combine, for the first time, not only methods useful in this field and applicable to a broad range of materials, but also considerable appreciation of the economics of the problem.

Requests have recently been received from Committee B-7 on Light Metals and Alloys for help in developing methods for determining color and gloss of base and anodized wrought aluminum alloy surfaces, and from Committee D-19 on Industrial Water for review of their proposed method of turbidity of water.

The officers elected for the ensuing term of two years are as follows:

Chairman, George W. Ingle.
Vice-Chairman, W. J. Kiernan.
Secretary, Richard S. Hunter.

STANDARD CONTINUED WITHOUT REVISION

The committee recommends the continuation without revision of the Standard Method of Test for 45-deg, 0-deg Directional Reflectance of Opaque Specimens by Filter Photometry (**E 97 – 55**).[1]

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* Sixty-first Annual Meeting of the Society, June 22–27, 1958.

[1] 1955 Book of ASTM Standards, Parts 3, 4, and 7.

This report has been submitted to letter ballot of the committee, which consists of 78 members; 34 members returned their ballots, all of whom have voted affirmatively.

Respectfully submitted on behalf of the committee,

GEORGE W. INGLE,
Acting Chairman.

RICHARD S. HUNTER,
Secretary.

REPORT OF COMMITTEE E-13
ON
ABSORPTION SPECTROSCOPY*

Committee E-13 on Absorption Spectroscopy, together with most of its subcommittees, met in Pittsburgh, Pa., on March 3, 1958. This meeting was held in conjunction with the Pittsburgh Conference on Analytical Chemistry and Applied Spectroscopy. The Advisory Committee met on September 11, 1957, in New York City and on March 2, 1958, in Pittsburgh.

The officers elected for the ensuing term of two years are as follows:

Chairman, E. J. Rosenbaum.

Vice-Chairman, L. E. Kuentzel.

Secretary, R. F. Robey.

Advisory Committee Members-at-Large, W. C. Kenyon and J. M. Vandenbelt.

During the year Subcommittee VI on Fluorescence Spectroscopy (R. L. Bowman, chairman) was established to organize activity in this resurgent field. Since magnetic resonance spectroscopy is deemed within the present scope of Committee E-13, a task group is being organized.

Revised Scope.—The committee is recommending to the Board of Directors that its scope of activity be broadened better to accomplish the first of the two chartered purposes of the Society, namely, "promotion of knowledge of the materials of engineering." The recommended revised scope follows:

> The advancement of the fields of spectroscopy involving molecular, atomic, or nuclear absorption, and fluorescence, scattering, or polarization, by promoting exchange of information, by sponsoring meetings and symposia for presentation of papers, and by coordinating and formulating scientific practices and methods of analysis in these fields.

PROPOSED METHOD TO BE PUBLISHED AS INFORMATION

The committee recommends for publication as information only the Proposed Methods for Evaluation of Spectrophotometers as appended hereto.[1]

This recommendation has been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[2]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Apparatus Specifications (T. R. Harkins, chairman) prepared the Proposed Methods for Evaluation of Spectrophotometers referred to earlier in the report. The method covers (*1*) stray radiant energy, (*2*) spectral resolution, (*3*) wavelength accuracy, (*4*) wavelength repeatability, (*5*) photometric linearity and repeatability, and (*6*) absorption cells. Further review of the methods prior to submittal for publication as tentative is planned.

Subcommittee II on Methods (R. T. O'Connor, chairman) has prepared a Suggested Format for ASTM Methods

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* Sixty-first Annual Meeting of the Society, June 22–27, 1958.

[1] See p. 472.

[2] The letter ballot vote on this recommendation was favorable; the results of the vote are on record at ASTM Headquarters.

Involving Absorption Spectroscopy. Although the format has been approved by letter ballot of the main committee, it is being withheld from publication until two recommended practices to which it refers are approved and published. These practices cover general techniques of infrared and ultraviolet quantitative analysis. Preparation of a Suggested Practice for Identification of Materials by the Kuentzel Absorption-Spectral Method is planned. This will refer to the Wyandotte-ASTM Punched-Card Index of Absorption Spectral Data.[2]

Subcommittee III on Standard Data (M. V. Otis, chairman) with the aid of volunteer workers coded about 3000 infrared and 1200 ultraviolet-visible spectra during 1957. Only published spectra are coded. When these codes are punched into cards, the spectral index will comprise about 15,000 infrared and 12,000 ultraviolet-visible cards. In addition, a 20,000-unit Formula-Name Index to the infrared cards is available, and similar coverage of the ultraviolet cards is planned. Cards for indexing of spectral data in the near-infrared range (the range of spectrum between the visible and the conventional infrared) are being drawn up.

Subcommittee IV on Nomenclature (W. P. Tyler, chairman) plans to extend the list of Tentative Definitions of Terms and Symbols Relating to Absorption Spectroscopy (E 131 - 57 T). The terms and symbols used in the Proposed Methods for Evaluation of Spectrophotometers have priority in this project.

Subcommittee V on Publications (M. G. Mellon, chairman) has edited the Proposed Methods for Evaluation of Spectrophotometers.

Subcommittee VI on Fluorescence Spectroscopy (R. L. Bowman, chairman) plans to sponsor a symposium in its field in conjunction with the meeting of the Society for Applied Spectroscopy, November 6 and 7, 1958, in New York City. Such a symposium will serve as a starting point for further organization of the subcommittee along lines of apparatus specifications, methods, presentation of data, and nomenclature.

This report has been submitted to letter ballot of the committee, which consists of 55 members; 55 members returned their ballots, of whom 52 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

E. J. ROSENBAUM,
Chairman.

R. F. ROBEY,
Secretary.

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[2] Available at ASTM Headquarters.

APPENDIX

PROPOSED METHODS FOR EVALUATION OF SPECTROPHOTOMETERS[1, 2]

These are proposed methods and are published as information only. Comments are solicited and should be addressed to the American Society for Testing Materials, 1916 Race St., Philadelphia 3, Pa.

Scope

1. (*a*) These methods are intended for performance evaluation of spectrophotometers as a means of selection of apparatus suitable for specific test methods. To avoid specifying instruments supplied by particular manufacturers, objective specifications of instrumental performance have been written.

(*b*) A study of those instrumental properties which have a significant effect on the quality of analytical results obtained has been made, and a group of methods for measuring these properties has been developed. The evaluation of these properties will make it possible for each ASTM spectrophotometric method to be written with a statement of the limiting values of the significant properties that an instrument must have in order to be considered adequate for use in that method.

(*c*) The following topics were selected as significant: wavelength repeatability, wavelength accuracy, spectral resolution, photometric repeatability and linearity, stray light, and absorption cells. Each of these topics is considered in turn in a separate part of the methods. In general, each part presupposes that the tests of the preceding part have been applied.

PART I.—WAVELENGTH REPEATABILITY

Introduction

2. (*a*) Lack of repeatability of wavelength readings occurs when the radiant power that emerges from a monochromator differs in wavelength from time to time, even though the monochromator is set to the same nominal wavelength. The main causes of this lack of repeatability are changes in temperature of various parts of the monochromator, particularly the crystal prism, and lack of repeatability of the position of various parts of the system. In recording spectrophotometers, a change in response characteristics of the electrical system will also show up as apparent changes in wavelength.

(*b*) When recording charts with printed wavelength scales are used, apparent wavelength errors may be due to errors in positioning the chart on the recording drum. Successive batches of purchased chart paper should be checked for uniformity of chart spectrum length, particularly if the paper has been subjected to pronounced humidity changes.

(*c*) Although the effect of a variation in temperature on the index of refraction of a prism can be found readily, the relationship between ambient temperature change and prism temperature change depends on the design of the instrument. Thermostatting can reduce the effects of ambient changes, and

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[1] These proposed methods are under the jurisdiction of the ASTM Committee E-13 on Absorption Spectroscopy.

[2] Published as information, June, 1958.

bimetallic elements can be used to compensate for the wavelength changes accompanying differences in temperature. In cases where most accurate readings are desired, operation in a temperature controlled room may be necessary.

(*d*) Looseness or backlash in the system leads to a lack of mechanical repeatability, but this can be overcome if proper conditions of operation are used. Other mechanical effects, such as changes in friction, changes in the thickness of oil films, or the amount of dirt or dust between contacting surfaces, are random effects and cause erratic behavior.

(*e*) In recording spectrophotometers there is always some lag between the recorded reading and the correct reading. If the scan is slow, and the response of the system is sufficiently fast, this effect is small. If the conditions of operation are the same for repeated runs, then the effect should be repeatable. If the scan rate is quite high, however, measurements made at the same settings and with the same apparent conditions of operation will not give truly repeatable results. For this reason, *when calibrating recording spectrophotometers, the same instrumental settings should be used as during actual measurements.*

Measurement Procedure

3. (*a*) *General.*—Conditions under which the test is run should match the conditions that a prudent operator would use for most of his measurements. For example, when the wavelength of a single absorption band is to be measured repeatedly, the instrument should be started far enough behind the position of the band so that any backlash in the system is taken up. Similarly, with a recording instrument, the controls for scanning speed, amplifier gain, response time, and slit width should be set for normal operating ranges, and the same nominal settings should be used for each run. Position the recording chart paper carefully.

To test ambient temperature effects two tests should be used. In the first, the temperature range should be varied widely, and the effect on the wavelengths of the bands should be measured. In the second, the wavelengths should be measured several times in a single day to determine whether the wavelengths are shifted as the instrument gradually warms up or the temperature of the room changes.

For detecting mechanical troubles, a number of successive, repeat measurements are usually sufficient.

(*b*) *Ultraviolet and Visible Regions:*

(*1*) *Mechanical Repeatability.*—Allow the instrument to come to a steady state temperature. Measure the wavelengths of the lines of a suitable arc source or absorbing sample at intervals during a day. A number of suitable, fixed wavelength reference points (**1**)[3] in various regions of the spectrum are given in Part II on Wavelength Accuracy.

(*2*) *Temperature Repeatability.*—By bringing the instrument to constant temperature, as recommended in Item (*1*), the effect of temperature can be eliminated. In determining temperature repeatability, mechanical effects cannot be completely eliminated. Under the best operating conditions there will be slight mechanical variations.

If the room temperature is *constant* within 2 to 3 C, turn the instrument on for the warm-up period recommended by the manufacturer. Observing the precautions for mechanical repeatability (except, of course, that of a steady temperature), the wavelengths of three suitable lines should be recorded. This recording should be repeated hourly during the day to see how long the instrument must be on before there is no shift of wavelength with temperature.

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[3] The boldface numbers in parentheses refer to the list of references appended to these methods, see p. 494.

If the room temperature *varies* more than 2 to 3 C, follow the same instructions for operating in a room at constant temperature. In addition, keep a record of the variation of the room temperature in the vicinity of the instrument during the day, and determine the wavelengths of three suitable lines as a function of temperature.

(*c*) *Infrared Region.*—Determine mechanical repeatability and temperature effects by the procedures described in Paragraph (*b*). Use toluene as the sample in a 0.02- to 0.03-mm sealed liquid cell. Obtain its spectrum in the regions 3.2 to 3.4, 9.1 to 9.4, and 14.2 to 14.6 μ. Locate the central absorption band in each region. For each band note the approximate transmittance at the minimum and draw a line on the spectrum at a transmittance level 10 per cent higher than the minimum. Measure the wavelength of the intersections of this line with the two sides of the absorption band, and average these two values to find the observed wavelength of the band. Repeat each measurement ten times, average the observed wavelengths, and calculate the average departure from the mean value. The correct positions of the toluene bands are 3.29, 9.25, and 14.42 μ.

PART II.—WAVELENGTH ACCURACY

Introduction

4. (*a*) In making measurements on overlapping absorption bands, it is important to locate spectral positions accurately. This is particularly true if measurements must be made at the sides of bands.

(*b*) Because wavelength scales can get out of adjustment easily, frequent, even daily, checking is advisable. The details of this check depend on the spectral range of interest and the type of instrument in use. If serious errors are found, the manufacturer's literature should be consulted for instructions.

(*c*) The precautions of Part I on Wavelength Repeatability are important in properly checking the wavelength scale. Wavelength readings change so much with temperature that the instrument must be turned on long enough to allow a steady instrument temperature to be reached. Calibration of a recording spectrophotometer may also be affected by positioning of the sheet, and by changes in size of the paper with temperature and humidity.

Ultraviolet Region

5. (*a*) *Use of an Arc Source:*

(*1*) If the lamp supplied with the instrument can be changed for another source, the lamp should be replaced by a quartz mercury arc[4] or by a mercury arc that has an envelope transmitting in the ultraviolet[5] (Note 1). The arc should be properly aligned and focused on the entrance slit.

NOTE 1.—The use of the General Electric H85-C3 lamp makes it unnecessary to follow the dangerous practice of removing the outer glass envelope from the General Electric H100-A4 lamp in order to use the bare quartz capillary of this lamp. The ultraviolet output of the H85-C3 lamp is about the same as that of the bare quartz capillary of the H100-A4 lamp.

The General Electric germicidal lamp G4T4/1 produces much less continuous background than the high-pressure lamps and the line at 253.7 mμ is not reversed. For some purposes the other lines emitted by this lamp may not be sufficiently intense.

The General Electric mercury-cadmium lamp H100-F4 is also useful, since it gives a number of cadmium lines in regions where there are no mercury lines.

(*2*) If the spectrophotometer is nonrecording, one of the following calibration lines should be located: 237.83,

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[4] The General Electric type H85-C3 lamp has been found satisfactory for this purpose.

[5] The Beckman mercury arc conforms to this requirement.

253.65, 275.28, 296.73, 312.57, 334.15, and 365.01 mμ. Care should be taken to avoid confusion with other mercury lines that are present (Note 2).

NOTE 2.—K. S. Gibson (**1**) states that with a Beckman DU spectrophotometer the lines at 365.01, 365.48, and 366.33 should all be seen, the ratio of their intensities being approximately 100:48:36.

(*3*) Next, the wavelength drive should be adjusted carefully until a maximum apparent transmittance reading shows that an accurate setting on the line has been reached and the wavelength dial then read. All of the lines listed should be checked similarly.

(*4*) If a recording spectrophotometer is used, the positions of the lines listed above are read on the recorder chart. A relatively slow scanning speed should be used to make sure that the detecting system and recorder can follow the signal pulses resulting from narrow emission lines (Note 3).

NOTE 3.—A slowly responding system will displace all of the lines in the direction scanned. It may be necessary to know the amount of this displacement, if there is one, at "normal" speeds.

(*b*) *Use of an Absorbing Sample.*—No substance has been found having absorption bands that provide a satisfactory wavelength calibration in the ultraviolet. As a routine procedure it is advisable to use a dilute solution of benzene in *iso*octane (2,2,4-trimethylpentane) to check the position of the absorption band at 254.8 mμ.

Visible Region

6. (*a*) *Use of an Arc Source:*

(*1*) If the lamp supplied with the instrument can be changed for another source, the lamp should be replaced by a quartz mercury arc[4,5] (Note 1). The arc should be properly aligned and focused on the entrance slit.

(*2*) If the spectrophotometer is nonrecording, locate one of the following calibration lines: 365.01, 404.66, 435.83, 491.60, 546.07, and 577.0 mμ. If the last line is not resolved from its close neighbor, use 578.0 mμ for the blend. Other lines present in the mercury spectrum should not be confused with those listed.

(*3*) Next, the wavelength drive should be adjusted carefully until a maximum apparent transmittance reading shows that an accurate setting on the line has been reached and the wavelength dial then read. All of the listed lines should be checked similarly. If a hydrogen arc is available, the line at 656.28 mμ may be checked.

(*4*) If a recording spectrophotometer is used, the directions given in Section 5 (*a*) (*4*) should be followed.

(*b*) *Use of an Absorbing Sample.*—When the lamp supplied with the instrument cannot be replaced easily, a suitable piece of didymium glass,[6] about 3 mm thick, may be used as an absorbing sample. Bands at the following wavelengths should be located: 441.0, 528.7, 585.0, 684.8, and 743.5 mμ. As the apparent positions of these bands depend to some extent on the slit width used, the instrument should be calibrated as far as possible with slits at the same width at which it will normally be used. The values given above were obtained with the slit of the spectrometer set so that the spectral band width obtained was 10 mμ.

Near-Infrared Region

7. In the region of 0.6 to 2.6 μ, both arc sources and absorbing samples may be used (Note 4). The techniques of

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[6] Corning 1-60 (formerly 5120) didymium glass has been found satisfactory for this purpose. The National Bureau of Standards supplies this glass with a spectral curve and values of the wavelengths of the principal absorption bands.

measurement described in Section 5 should be employed.

NOTE 4.—Plyler and Peters (2) and Acquista and Plyler (3) give fourteen lines between 1 and 2.4 μ for the General Electric H100-A4 arc, and Plyler, Blaine, and Tidwell (4) give lines for mercury, neon, argon, krypton, and xenon sources. Absorption bands for didymium glass, polystyrene, carbon disulfide, and 1,2,4-trichlorobenzene are given by Plyler, Blaine, and Nowak (5).

Infrared Region

8. (*a*) Because there are no arc sources of spectral lines in the infrared (2.6 to 15 μ), an absorbing sample must be used. As the apparent position of a narrow absorption band depends on the spectral band width, the narrowest slit widths for which an adequate signal-to-noise ratio is obtained should be used. Detailed discussions of precise wavelength calibration can be found in the literature (2 to 6).

The most generally useful sample for quick routine checks is a film of polystyrene about 0.1 mm thick. The positions of the following sharp bands should be located: 3.422, 6.692, 9.724, and 11.035 μ (Note 5).

NOTE 5.—As the band at 3.3026 μ (3027.1 cm^{-1} (vacuum)) is somewhat sharper than the one at 3.422 μ (2921.5 cm^{-1} (vacuum)) it will, when resolved, give a somewhat more accurate check on the wavelength.

(*b*) As a routine procedure it is advisable to check the position of the atmospheric carbon dioxide band at 4.258 μ. This band is a doublet that may or may not be resolved. The value given is for the branch of longer wavelength when the band is resolved.

(*c*) Because of drifts due to temperature changes, it is advisable to check spectra intended for reference or publication several times a day at one point. Preferably this should be done at a low wavelength, as temperature effects are most apparent here.

PART III.—SPECTRAL RESOLUTION

Introduction

9. (*a*) Resolution is defined as the ratio $\lambda/\Delta\lambda$, where λ is the wavelength of the region being examined, and $\Delta\lambda$ is the separation of two absorption bands that can just be distinguished. Resolution can also be defined as $\nu/\Delta\nu$, where ν and $\Delta\nu$ refer to the wavenumber scale. For a given region of the spectrum, a specification of $\Delta\lambda$ or $\Delta\nu$ gives a sufficient indication of instrument resolution to use in designating the different conditions of resolution.

(*b*) Four separate conditions of resolution, designated condition A, condition B, condition C, and condition D have been specified for the ultraviolet, near infrared, and infrared (sodium chloride prism) regions of the spectrum. Condition A is the lowest resolution that would probably be used in an analytical procedure. Conditions B, C, and D show increasingly better resolution (Note 6).

In a particular analytical procedure, one of these conditions will be set as a minimum necessary to obtain results of a specified accuracy. It may be possible to use a spectrophotometer for a method of test if it meets a certain condition in a given wavelength range, even though it does not meet it in other wavelength ranges, provided the method of test makes use of the region where the instrument passes the test.

NOTE 6.—As instruments are improved, or as the need may be required in a particular analysis, conditions of still higher resolution may be designated by E, F, ...

Instrument Operating Conditions

10. (*a*) *General Precautions.*—All operating parameters must be set to the same values in both the resolution test and the analytical method. Such parameters include wavelength scanning speed, response time, amplifier gain, detector

bias, servo system gain settings, slit widths, and other relevant variables. Narrower slit widths may be used for the method of test, but not wider ones. In the conventional infrared region, for example, better resolution values can usually be obtained by taking a longer time to make the measurement. This is accomplished by narrowing the slits, increasing the amplifier gain to restore normal signal to the recorder (or to restore normal servo loop gain in double beam instruments), and then increasing the response period to reduce the noise to the desired value. The increased response period then requires slower scanning speeds for accurate photometric results. On meter-reading instruments, one must take a correspondingly longer time for the needle deflection to reach equilibrium. Therefore, if a certain resolution condition is obtained only with such operating settings, the analytical procedure must not be performed so that a shorter time per measurement is used.

(*b*) *Operating Conditions Required.*—In order to secure satisfactory results, the following operating conditions should be maintained: (*1*) the slits of the monochromator should be set to the desired widths, thus determining the energy level at which the detector must operate, (*2*) the gain of the signal amplifier or of the servo amplifier must be set to give the proper response or sensitivity, (*3*) the damping or time constant of the detector system must be set at the value necessary to give the desired signal to noise ratio, and (*4*) the scanning speed of the instrument should then be set at the fastest speed that does not distort the recorded spectra.

(*c*) *Noise.*—These resolution conditions should be obtained when the peak-to-peak noise is less than 0.5 per cent transmittance.

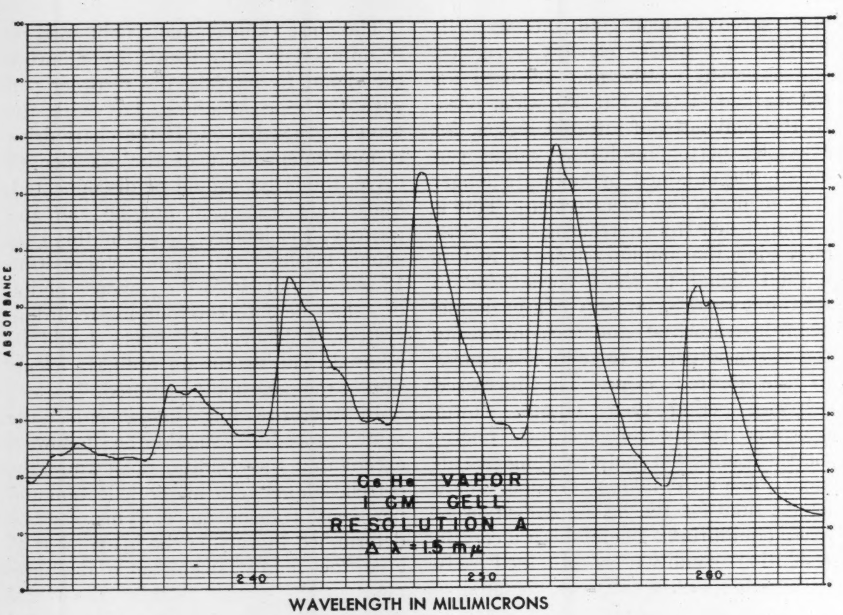
The noise value should be obtained on scanning-type instruments by allowing the signal to activate the recorder pen with the chart running, but with the wavelength drive off. If the chart drive is not independent of the wavelength drive, the latter should be disconnected. Since this test need not be run frequently, the inconvenience in this case should be slight. A time interval of 10 to 15 sec, or four times the response period of the detecting-amplifying-recording system, whichever is longer, should be allowed. The maximum ordinate excursion of the pen should be noted.

In meter-reading instruments, the same procedure should be followed, except that the operator must know the correlation between meter deflection and per cent transmittance. This can be determined by setting the instrument to give the final transmittance measurement of the sample, turning the transmittance dial through an additional 0.5 per cent transmittance, and noting the meter deflection.

Ultraviolet and Visible Region

11. (*a*) *Materials and Concentration.*—Benzene vapor in a 1-cm, stoppered silica cell shall be used. The benzene concentration must be such that the absorbance of the band at 236 mμ lies between 0.65 and 0.85. This concentration can usually be obtained by placing a few drops of liquid benzene in a 1-cm stoppered cell, allowing it to stand for 1 min, and then quickly emptying the cell and stoppering well.

(*b*) *Results.*—The four spectra of Fig. 1, show four conditions (A, B, C, and D) of resolution in the region of 236 mμ. To achieve a given condition, the instrument must resolve the peaks as well as shown on these spectra at the correct concentration of benzene.



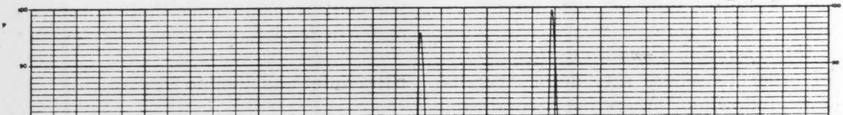
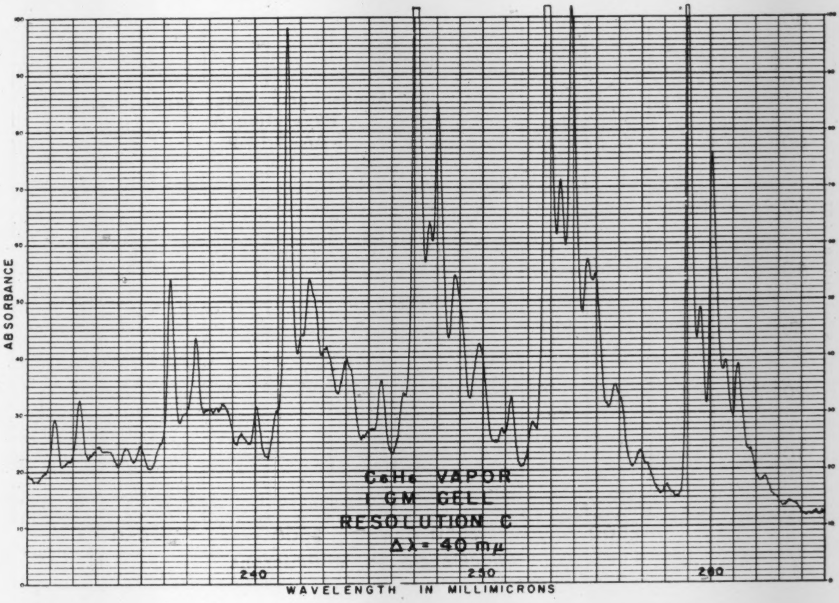
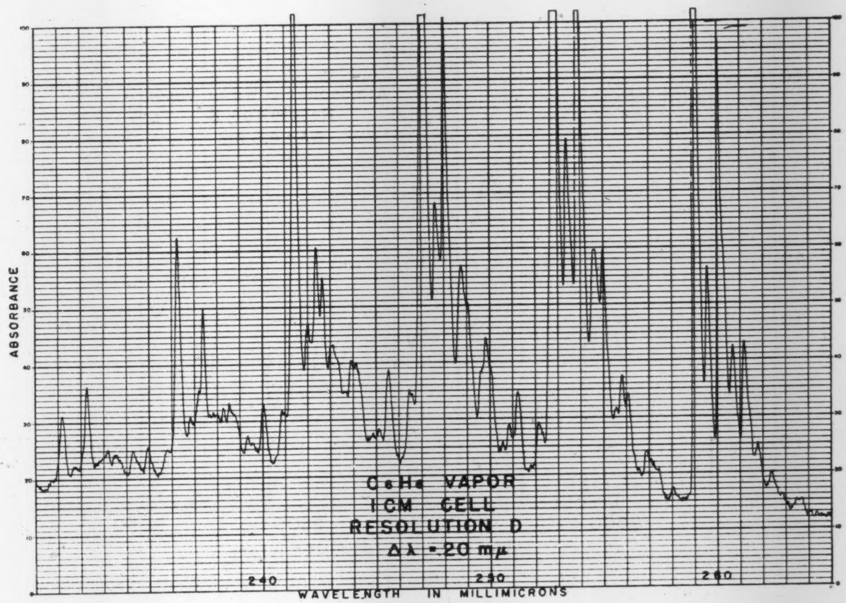


FIG. 1.—Absorption Spectra of Benzene Vapor Showing Four





Different Conditions of Resolution in the 236-mμ Region.

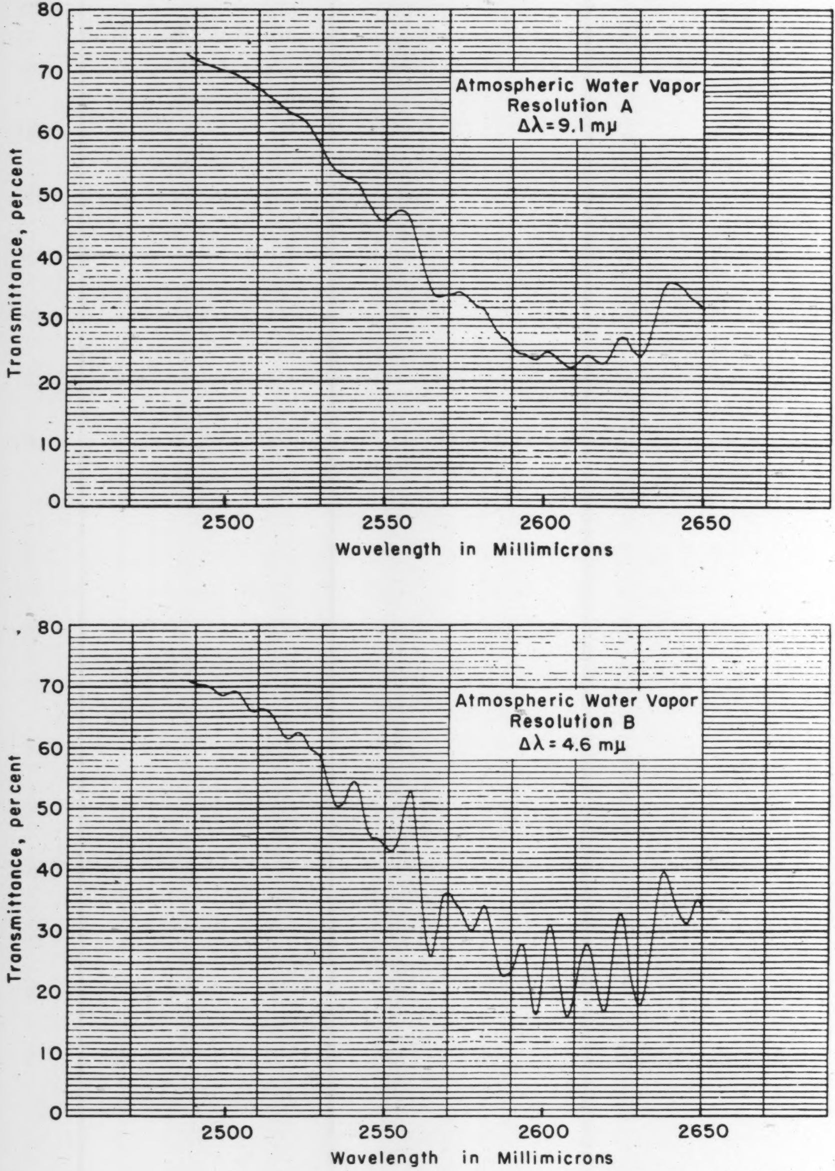
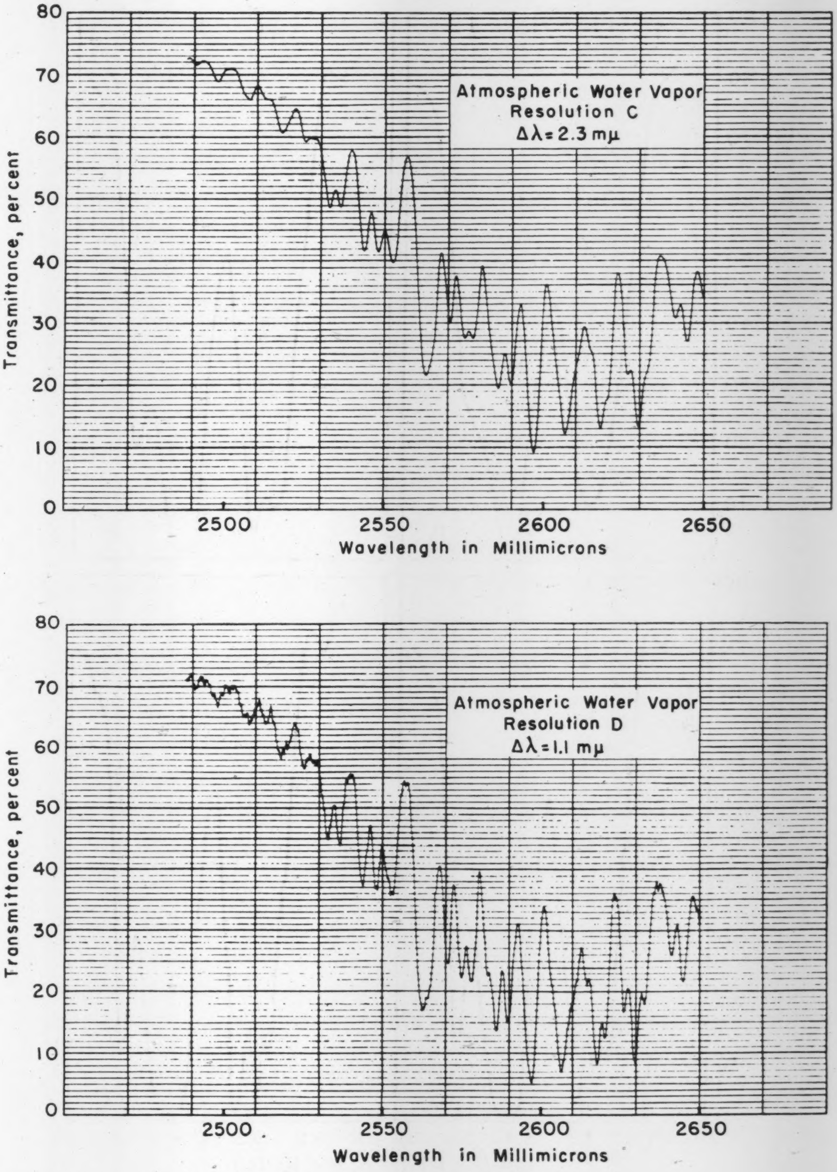


FIG. 2.—Absorption Spectra of Atmospheric Water Vapor Showing



Four Different Conditions of Resolution in the 2.6-μ Region.

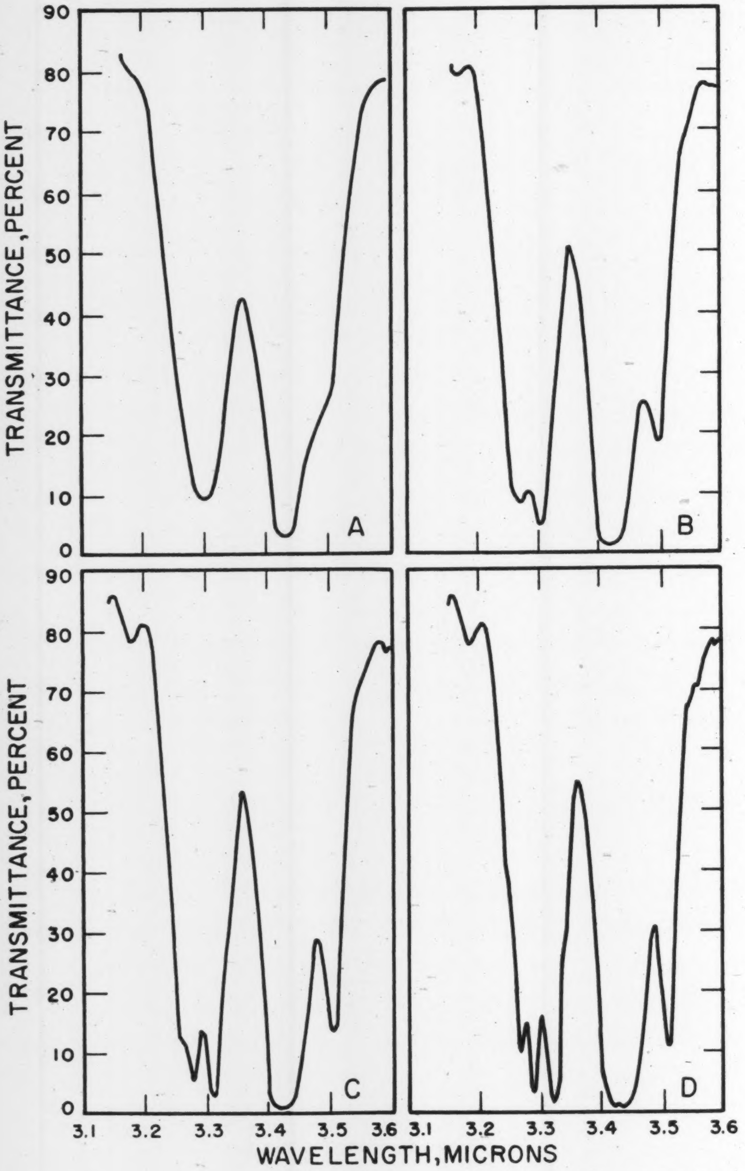


FIG. 3.—Absorption Spectra of Polystyrene Film Showing Four Conditions of Resolution in the 3-μ Region.

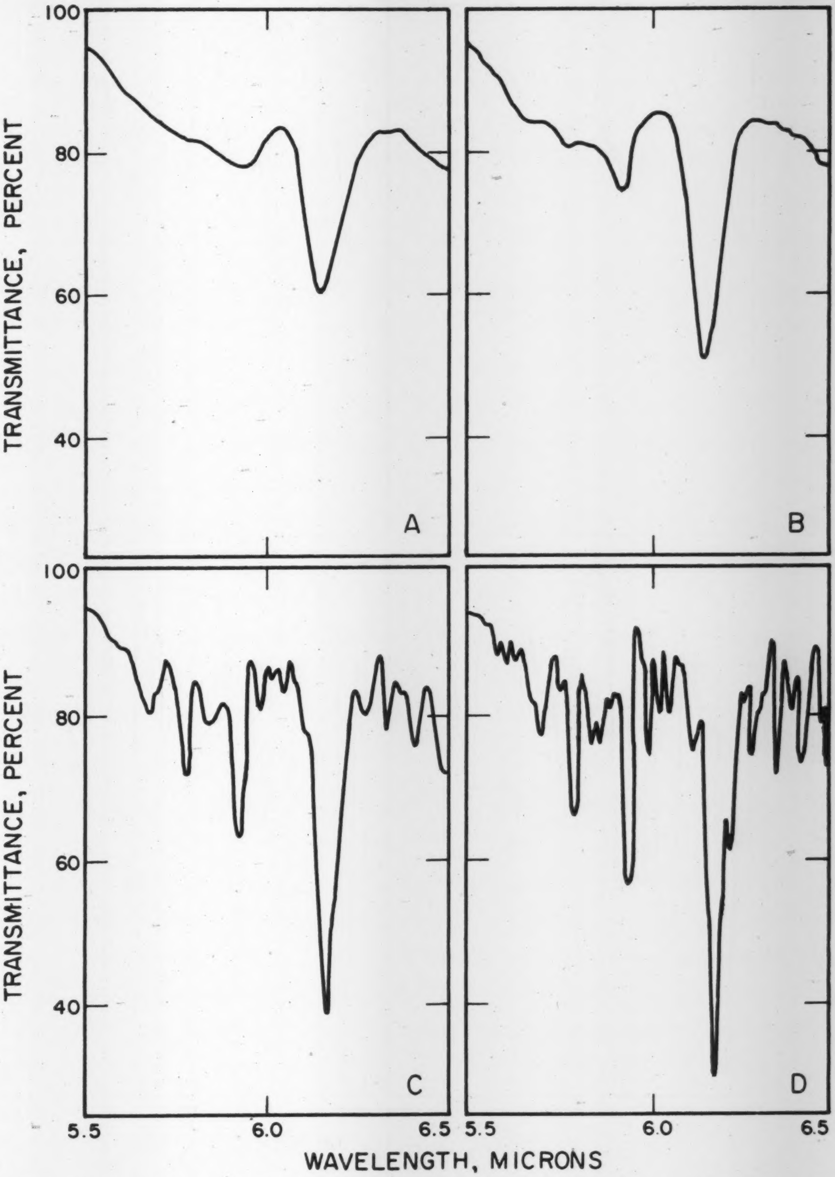


FIG. 4.—Absorption Spectra of Ammonia Vapor Showing Four Conditions of Resolution in the 6-μ Region.

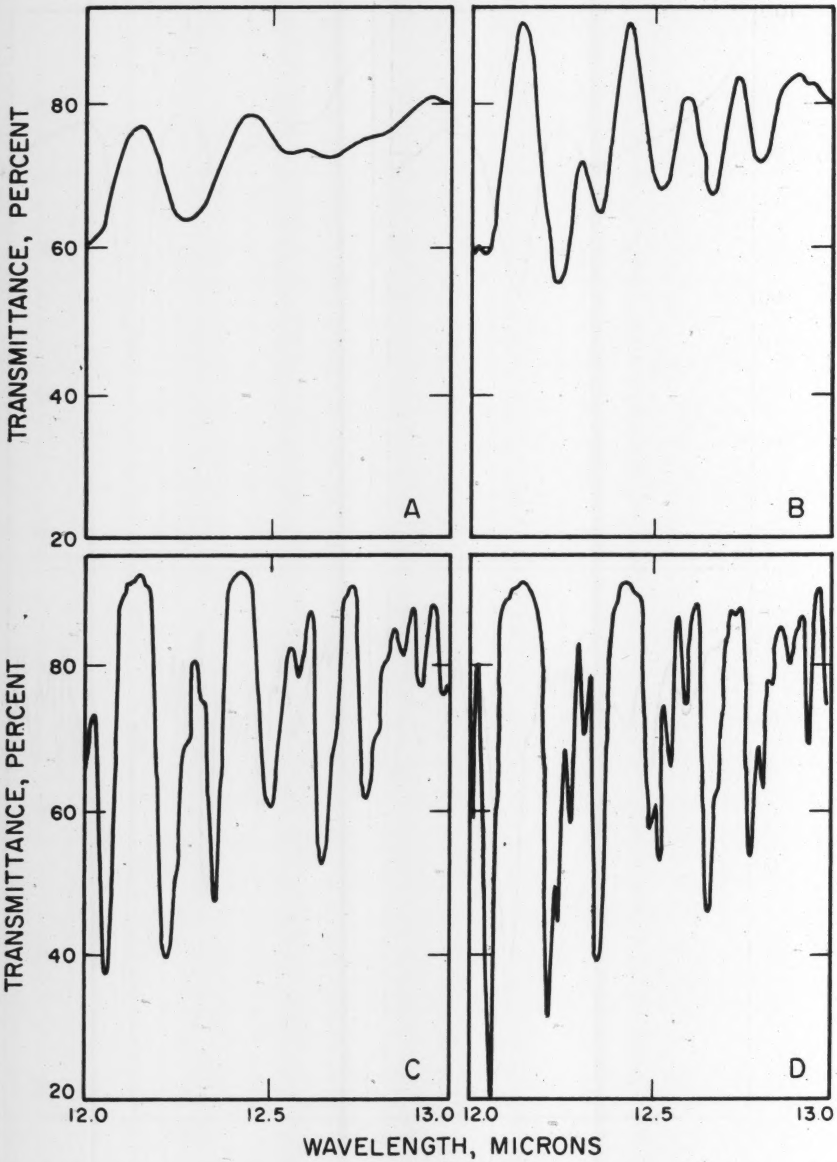


FIG. 5.—Absorption Spectra of Ammonia Vapor Showing Four Conditions of Resolution in the 12.5-μ Region.

Near-Infrared Region

12. (*a*) *Materials and Concentration.*—Atmospheric water vapor shall be used. Ordinary conditions of humidity will produce the proper concentration, unless the instrument is evacuated or purged with a dry gas. In the latter cases the instrument must be vented to the atmosphere.

(*b*) *Results.*—The four spectra of Fig. 2 show four conditions (A, B, C, and D) of resolution for water vapor in the region of 2.6 μ.

TABLE I.—RESOLUTION VALUES FOR DIFFERENT CONDITIONS.

Condition	Spectral Region			
		3 μ	6 μ	12 μ
A	$\lambda/\Delta\lambda$	20	40	80
	$\Delta\lambda$	0.15 μ	0.15 μ	0.15 μ
	$\Delta\nu$	170 cm^{-1}	40 cm^{-1}	10 cm^{-1}
B	$\lambda/\Delta\lambda$	40	80	160
	$\Delta\lambda$	0.075 μ	0.075 μ	0.075 μ
	$\Delta\nu$	83 cm^{-1}	20 cm^{-1}	5 cm^{-1}
C	$\lambda/\Delta\lambda$	100	200	400
	$\Delta\lambda$	0.03 μ	0.03 μ	0.03 μ
	$\Delta\nu$	34 cm^{-1}	83 cm^{-1}	2 cm^{-1}
D	$\lambda/\Delta\lambda$	200	400	800
	$\Delta\lambda$	0.015 μ	0.015 μ	0.015 μ
	$\Delta\nu$	17 cm^{-1}	4 cm^{-1}	1 cm^{-1}

Infrared Region (Sodium Chloride Prism)

13. (*a*) *Materials.*—For the test in the 3 μ region, a sheet of polystyrene 0.002 in. thick is necessary. Ammonia gas at 200 mm pressure in a 5-cm cell, or an equivalent concentration and path length, should be used for the 6 and 12 μ regions.

(*b*) *Results.*—Table I gives the values of resolution, $\Delta\lambda$ and $\Delta\nu$, for four conditions (A, B, C, and D) at the three test wavelengths. The spectra of Figs. 3, 4, and 5 show the resolution that should be obtained using the above materials for the four conditions specified.

PART IV.—PHOTOMETRIC REPEATABILITY AND LINEARITY

Introduction

14. (*a*) The basic requirements for the photometric system of a spectrophotometer are repeatability and linearity. Photometric linearity refers to the ability of a photometric system to yield a prescribed relationship between the radiant power incident on its detector and some measurable quantity provided by the system. In the case of a simple detector-amplifier combination, the relationship is direct proportionality between incident radiant power and the deflection of a meter needle or recorder pen. Photometric accuracy, although desirable, is not a basic requirement.

(*b*) Few valid techniques for determining photometric linearity exist. The method to be employed here depends on the agreement of instrumental results with Bouguer's law. This law states that the absorbance of a homogeneous sample is directly proportional to sample thickness. Tests of Bouguer's law in the visible, ultraviolet, and near-infrared regions (Note 7) are readily performed by using commercial filters which are interposed in the sample beam individually and in groups to determine whether the absorbance increments are additive. Accurate agreement between the sum of the absorbances of the individual filters and the absorbance of the group provides a high probability of photometric linearity.

NOTE 7.—At present this section covers test methods applicable to the ultraviolet, visible, and near-infrared spectral regions. The infrared region will be included at a later date. A section on photometric accuracy will also be added.

(*c*) For exacting tests of Bouguer's law there must be no beam deviations or multiple reflection effects to cause extraneous errors. This is accomplished by positioning each filter at a different angle to the beam to deviate the multiple-reflected rays, and substituting a transparent slide of equal optical thickness when a filter is removed from the beam to avoid beam deviations. A suitable angle in most spectrophotometers is 10 deg rotation about a vertical axis, giving a 20-deg deviation of the first reflected beam. In some spectrophotometers it may be necessary to lengthen the beam or to narrow the aperture in front of the phototube enough to avoid collecting the doubly reflected beam, or it may be necessary to do both.

(*d*) A still simpler arrangement is permissible if the beam in the cell space is collimated with respect to the photodetector. This provision makes the system insensitive to small lateral beam displacements. It is then not necessary to substitute a transparent slide for the removed filter.

(*e*) Metallic screens are available[7] with electrolytically perforated holes having conical cross-sections with sharp edges. Such screens are almost achromatic beam attenuators. If screens are precalibrated against standard filters or by measurement on the same spectrophotometer when it has been verified to be linear, they are recommended for routine use as secondary standards as well as for repeatability tests.

Precautions

15. (*a*) *Stray Radiant Energy.*—Stray radiant energy will cause photometric nonlinearity in tests that are sensitive to spectral composition, such as Bouguer's law tests with colored glass filters. Correction for the effects of stray radiant energy is not recommended because of the numerous and variable causes. The only safe assumption is that measurements are in error at least in proportion to the amount of stray radiant energy indicated by tests performed with the detector being tested. (See Section V on Stray Radiant Energy.)

(*b*) *Wavelength Dependence.*—A photometer that is linear at one wavelength is usually equally good elsewhere. Judgment must be exercised in applying this rule, however, as exceptions are known to exist. Only if tests giving concordant results bracket the wavelength region of interest is it safe to assume that the rule applies.

(*c*) *Photometer Response Characteristics.*—Preliminary tests should be performed to evaluate backlash and sticking friction of the photometer indicating recording device, to insure that good adjustment prevails and that results are not invalidated by readily prevented factors. Tests should include observation of indicator speed and hysteresis characteristics following both large and small excursions. For best accuracy, to avoid errors caused by paper guidance mechanisms and printing faults, results should be read from recorder fixed scales rather than charts.

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[7] Suitable screens are available from the Pyramid Screen Co., 264 Mason Terrace, Brookline 46, Mass. Screen 60M is fine mesh, with absorbance approximately 0.68. Numbers 15M and 10S are coarser, with absorbance approximately 0.36 and 1.1, respectively. Conical perforations in nickel sheet should be specified.

(*d*) *Sample Position.*—In order to be sure that the inevitable slight sample and beam nonuniformities do not affect results, samples must be marked and mounted to permit reproducing their position in the beam exactly. Optimum positions, if a choice exists, are those where the beam has best uniformity and largest cross-section area.

(*e*) *Effects of Polarization.*—The radiant energy passing most monochromators becomes strongly polarized. Detectors and their associated optical elements are seldom completely indifferent to the polarization state of the incident radiant energy. Hence, measurements of optically active or birefringent samples must be viewed with suspicion even when using an otherwise ideal photometer system.

(*f*) *Effects of Fluorescence.*—The effects of fluorescence in samples interposed between monochromator and detector may produce serious errors. Commercial spectrophotometers differ in sensitivity to fluorescence. "Sharp cut" filters[8] are useful to detect fluorescence effects.

TABLE II.—TRANSMISSION DATA FOR FILTERS.

Filter	Thickness	Wavelength, $m\mu$	Approximate Absorbance	Maximum Bandwidth,[a] $m\mu$
Corning 5900	1.6 mm	410	0.12 (min)	8
		565	0.30 (min)	11
		655	0.45 (max)	7
		695	0.41 (min)	15
		820	0.45 (max)	35
Corning 3307	1.2 mm	350	0.55 (min)	5
		410	0.88 (max)	7
		500	0.36 (side)	16
		660	0.07 (min)	50
		1200	0.14 (max)	150
Corning 9782 (4-96)	Standard	500	0.16 (min)	11
		2200	0.45 (max)	20
		2420	0.38 (min)	30
Corning 7910	Standard	230	0.57 (side)	14
		240	0.36 (side)	65
Corning 5120 (1-60) Didymium	Standard	1510	0.43 (max)	75
		1900	0.32 (max)	20
		2380	0.32 (max)	10
		2950	0.36	30

[a] Resolution required for absorbance measurement accuracy to ±0.001. Resolution is specified in "effective band width" = "½ spectral band width" (E. F. Hartree, *Bulletin*, British Photoelectric Spectrometry Group, Vol. 1 (1949)).

Apparatus

16. (*a*) *Screens.*—Screens[7] should be mounted in holders similar to those used for glass filters (see Paragraph (*b*)).

(*b*) *Tests of Bouguer's Law.*—Suitable glass filters for use in these tests, together with recommended wavelength points and approximate respective absorbance values, are given in Table II. Thicknesses are chosen to provide a wide selection of absorbance values occurring at maxima and minima. Those filters with thicknesses which differ from Corning standards are purchased as molded plates, ground and polished to meet the following specifications: Select areas free of striae and large bubbles. Finish to spectacle lens quality, flat to ten fringes to within $\frac{1}{16}$ in. of edges, and parallel to within 6 min of arc.

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[8] "Glass Color Filters," Corning Glass Works, Optical Sales Dept., Corning, N. Y., have been found satisfactory for this purpose.

To protect filters from contamination and scratches and to secure them reproducibly in the beam, it is recommended that they be mounted in blackened aluminum or brass frames. The mount should secure the filter by spring tension or otherwise to prevent motion and should center the filter in the beam.

Use of filters in sets of three, mounted normal and at plus and minus ten degrees from normal to the beam, is recommended.

Measurement Procedure

17. (*a*) *Screen Checks for Repeatability:*

(*1*) Set the spectrophotometer controls to duplicate the conditions of intended analytical application.

(*2*) Measure the screen transmittance compared with air path.

(*3*) Compare with the initial check value.

(*b*) *Test of Photometric Linearity:*

(*1*) Adjust the spectrophotometer to duplicate conditions typical of analytical use. See Table II for wavelengths and slit width limits for recommended filters.

(*2*) If operating at the maximum or minimum absorption of the filter, set the wavelength dial for the exact wavelength of the peak.

(*3*) Record the operating conditions.

(*4*) Record the transmittance or the absorbance for each filter separately, then in pairs, trios, etc., in all combinations.

(*5*) To calculate the error, divide the products of the individual transmittances into the transmittance of the combinations, or subtract the sum of the individual absorbances from the absorbance of the combinations.

PART V.—STRAY RADIANT ENERGY

Introduction

18. (*a*) Stray radiant energy is all radiant energy reaching the detector at wavelengths different from the nominal wavelength band for which the instrument is set. It is commonly expressed as a percentage of the total energy or power (I_o or P_o) reaching the detector in the absence of an absorbing sample.

(*b*) "Near" stray radiant energy is that component of the total stray radiant energy which has wavelengths near that of the nominal wavelength. Although not covered specifically with a test method, it may be important separately in the use of exceptionally sharp, narrow bands, at any wavelength. A discussion of the effects of near stray radiant energy, and of a method of measurement in the ultraviolet, has been presented elsewhere (7).

(*c*) Stray radiant energy causes observed absorbance values to differ from the true values. Usually the observed values will be lower, but they may be the same or higher (8). Stray radiant energy is one of the factors leading to curves of concentration *versus* absorbance that are not straight lines at high values of absorbance. It may cause error in the calculation of absorptivities. The accuracy of differential measurements may be decreased by stray radiant energy, since errors are greatest at high absorbance.

Specification of Stray Radiant Energy

19. (*a*) *General.*—The amount of stray radiant energy is expressed as that fraction of the total radiant energy reaching

the detector which has wavelengths other than the nominal wavelength band and within the range of sensitivity of the detector. It is only this fraction which affects the observed value of the transmittance. A more exact specification would involve not only the amount but also the distribution curve of the radiant energy reaching the detector, emphasis being given to the wavelength region of high detector sensitivity.

TABLE III.—CONDITIONS UNDER WHICH STRAY RADIANT ENERGY MAY INTRODUCE SERIOUS ERRORS.

Wavelength Region, μ	Optics	Source	Detector
0.240....	quartz	UV	UV
0.350....	quartz	tungsten	UV
	glass	any	UV
	any[a]	any	visible
0.650....	quartz or glass	tungsten	UV or visible
2.5......	any	any	PbS cell
12........	NaCl	any	any
20........	KBr	any	any
30........	CsBr	any	any
35........	CsI	any	any

[a] Applicable for the region indicated.

Stray radiant energy may be estimated by comparing the *observed* transmittance with the *true* transmittance of samples that have very low transmittance values at the wavelength of measurement but are transparent elsewhere. The fraction of incident radiant energy that is stray is approximately the difference between observed transmittance and true transmittance:

$$T_s = \text{stray radiant energy fraction} = T\text{ (observed)} - T\text{ (true)}$$

There is normally a definite relationship between the stray radiant energy at one wavelength and the values at all other wavelengths where the same instrument, source, and detector are used. Thus, it is possible to characterize the behavior of an instrument, source, and

TABLE IV.—SUBSTANCES RECOMMENDED FOR DETERMINING STRAY RADIANT ENERGY.

Wavelength, μ	Sample	True Transmittance
0.205...	Corning Vycor filter No. 7910, 3.3 ±0.3 mm thick,[b] polished both sides	10^{-6}
0.310...	Corning filter No. 9782, 5.0 ± 0.1 mm thick,[c] polished both sides	10^{-4}
0.750...	Corning filter No. 9782, 5.0 ± 0.1 mm thick, polished both sides	10^{-5}
2.72[a]...	Corning Vycor filter No. 7910, 3.3 ± 0.3 mm thick, polished both sides	10^{-3}
9.00....	Lithium fluoride plate, 5.0 mm −0+2 mm thick, polished both sides	10^{-3}
14.3[a]....	Polystyrene sheet.[d] Transmittance of band at 11.0 μ shall be 31 ± 5 per cent *T*	10^{-3}
25.00....	Sodium chloride plate,[e] 5.0 mm −0+2 mm thick, polished both sides	10^{-3}
35.00..	Potassium bromide plate,[e] 5.0 mm −0+2 mm thick, polished both sides	10^{-3}
49.00....	Cesium bromide plate,[e] 5.0 mm −0+2 mm thick, polished both sides	10^{-3}

[a] Minimum of band.

[b] The standard Corning polished thickness of 2.0 ± 0.1 mm for the 7910 Vycor filter is not thick enough for satisfactory near infrared measurement. A thickness of 3.3 mm is satisfactory and can be obtained at a moderate increase in price (A 2 in. square piece costs about $5.00). If the piece is made significantly thinner than the nominal value, there will be some true transmittance which will be measured as stray radiant energy. If it is made significantly thicker, the measured transmittance will be too low (compared with the true stray radiant energy) due to absorption at other wavelengths.

[c] The 9782 filter has a standard polished thickness of 5.0 ± 0.1 mm. It is a standard Corning thickness and may be obtained at no additional charge.

[d] Lots of polystyrene at nominal 0.002 in. thickness usually fall within the specifications. Some manufacturers give away this material for advertising.

[e] The alkali halide standards suggested are rather expensive. A tolerance up to 2 mm thickness allows for samples already in existence. If tolerances much larger than these are used, the standard would lose its validity, due to the differences in readings obtainable between two standards both of which are within tolerance.

detector over their complete wavelength range by a measurement at one wavelength in that range, to the degree of accuracy required for the purposes of the particular test method. This is best done at an extreme point of the range, where the percentage of stray radiant energy is greatest.

A method requiring a measurement at 220 mμ might require that the stray radiant energy be less than 1 per cent near the extreme point of the range, say 205 mμ. This can be represented as: T_s (205) $<$ 1 per cent. In general, the stray radiant energy allowable can be specified at any wavelength as follows: T_s (λ) $<$ n per cent.

(*b*) *Conditions Under Which Stray Radiant Energy May Be Significant.*—Usually stray radiant energy is particularly important under the conditions listed in Table III. Under other conditions, the effects can frequently be neglected. Any absorbance value greater than 1.3, at any wavelength, is a critical absorbance condition.

Measurement Procedure

20. In order to determine the stray radiant energy proceed as follows: operate the instrument in the same way as when using it in an analytical method requiring the measurement, and measure the transmittance of an appropriate filter or sample listed in Table IV for the wavelength specified. Wavelengths must be accurate to ±2 per cent (Note 8). The value of the transmittance obtained is a measure of the stray radiant energy, T_s (λ) (Note 9).

NOTE 8.—The rather large tolerance for wavelength is allowable because of the opacity of the samples near the wavelength specified.

Since the true transmittances of all samples and filters shown in Table IV are negligibly small at the indicated wavelengths, the true transmittance term may be disregarded, and only the observed reading need be recorded.

NOTE 9.—One possible approach is to estimate the stray radiant energy in an instrument at the wavelength to be used in the analysis by first increasing the concentration or sample path length of the material absorbing at that wavelength until the true transmittance is very low, say 0.01 per cent, as predicted by Beer's law or the calibration curve. Then T_s (analytical λ) is measured in a manner similar to that given in Section 20, for the standard wavelengths, but using the sample and wavelength described above. The value T_s (standard λ) is measured at the appropriate standard wavelength, using the filter as described in Section 20. If it is determined that a value of N per cent stray radiant energy is permissible at the analytical wavelength, then the tolerance at the standard wavelength may be established as follows:

$$N(\text{standard }\lambda) = N(\text{analytical }\lambda) \times \frac{T_s(\text{standard }\lambda)}{T_s(\text{analytical }\lambda)}$$

PART VI.—ABSORPTION CELLS

Introduction

21. There are a number of cell window materials that can be used in the various regions of the spectrum. Some pertinent information concerning the more common materials is given in Table V. No attempt will be made to discuss the individual materials in detail. For such a coverage, reference should be made to the literature (9,10).

Ultraviolet, Visible, and Near-Infrared Cells

22. (*a*) *General Considerations.*—The most common cell used in this spectral region is the 1-cm path length liquid cell. In addition, the 5- and 10-cm path length cells are quite common. These cells have optical windows of either fused silica or glass. The useful wavelength ranges for each of these materials is given in Table V.

Ideally a cell should serve only as a holder for the sample and not contribute anything of its own to the sample's absorption. Practically speaking, all materials have some absorbance and the requirement becomes one of constancy and uniformity. Cells should be optically clear and have windows that are parallel to each other and perpendicular to the light path. In all analytical methods, the absorbance of different solutions is to be compared. With most instruments, several cells are used and therefore it is important to have cells that are matched to each other. The tests to be applied are designed to demonstrate the uniformity of the cells and to determine the correction factors to be applied if small differences are observed. For precise work, since there are usually small differences between cells, the cells should always be positioned in the same way in the holder; and the holder should, of course, always be positioned in the same way in the instrument.

TABLE V.—CELL WINDOW MATERIALS.

Material	Resistance to Water Vapor	Recommended Range, μ	Useable Range, μ
Glass	excellent	0.35 to 2	0.35 to 2
Fused silica	excellent	0.22 to 3.5	0.2 to 4
Crystal quartz	excellent	0.19 to 3.5	0.19 to 4
Sapphire	excellent	0.7 to 5.5	0.7 to 6
CaF_2 (fluorite)	good	0.19 to 9	0.19 to 10
BaF_2	excellent	0.19 to 12	0.19 to 14
NaCl	fair	2 to 15	0.2 to 17
KBr	poor	12 to 25	0.2 to 25
Silver chloride	good	0.6 to 22	0.6 to 25
KRS-5 (thallium-bromo-iodide)	good	15 to 40	0.6 to 40
CsBr	very poor	15 to 40	0.3 to 40

The most common cause for marked differences between absorption cells is dirty optical windows. If cells are not properly rinsed or if the rinsing solution leaves a residue on evaporation, a film may be formed on the optical window that absorbs part of the radiation. Distilled water or reagent grade methanol are suitable solvents for rinsing cells. When handling, care should be taken to avoid touching the optical windows and the cell should be wiped clean before placing in the cell holder. If cells become dirty they can be cleaned by soaking in water or a mild sulfonic detergent. If residue persists, a mixture of equal volumes of 3*N* HCl and alcohol has been recommended. For fused silica cells soaking in chromic acid cleaning solution is used by many, but care must be taken to rinse the cells with water immediately after removing from the cleaning solution. Alkaline solutions, detergents containing "optical bleaches," abrasive powders, fluorides, and materials that might etch the optical windows should not be used.

When measurements are made at wavelengths less than 220 mμ, special care must be taken to avoid errors due to fluorescent emission from the cell windows and polarization in the case of crystal quartz windows. A summary of the problems associated with measurements at these short wavelengths and some suggestions for solving them are given in the literature (**11**).

A knowledge of the absolute path length of a cell is not essential in analytical procedures where similar solutions are only compared. However, when determining absorptivities or some related value the path length enters into the calculation and must be known. An accurate determination of the path length is not practical in most laboratories and the best practice is to purchase a cell of known path length.

(*b*) *Measurement Procedures:*

(*1*) *Cleanliness.*—Determine the absorbance of the cell *versus* air reference. This procedure primarily tests for cleanliness, although gross differences in thickness or parallelism of the optical windows will also be detected. Fill the cell with clean, distilled water. Measure its absorbance against air at 240 mμ for quartz cells and 650 mμ for glass cells. With recording instruments, it is desirable to scan over the spectral region of interest. Rotate the cell in its holder (180 deg) and measure its absorbance again. The absorbance should be in the range of 0.083 to 0.093 for 1-cm quartz cells and 0.025 to 0.035 for 1-cm glass cells. Rotating the cell in the holder should not give an absorbance difference greater than 0.005.

(*2*) *Matched Cell Windows.*—This test compares the thickness of the optical windows and to a lesser extent the difference in path length. Fill both cells with clean, distilled water. Determine the absorbance of one cell as compared to the other cell as reference at the wavelengths to be used in the analytical procedure. The difference in absorbance should be less than 0.003 for matched cells. Differences greater than 0.015 usually indicate dirty cells. Cells that do not meet the above specification may be used after determining the difference in absorbance between the cells using the appropriate reference solution in both cells at the wavelengths of interest and applying this difference as a correction factor to the absorbances measured in that cell.

(*3*) *Matched Path Length.*—Prepare a solution of 0.001 *M* sodium dichromate ($Na_2Cr_2O_7$) in 0.01 *N* sulfuric acid (H_2SO_4). Filter if not absolutely clear. In a 1-cm cell this solution should have an absorbance of about 0.5 in the region of 430 mμ. Rinse the cells to be compared with the solution and then fill. Measure their absorbance in the region of 430 mμ as compared with distilled water as reference. Repeat this procedure. The difference between the average absorbance values for the cells, corrected for differences in blank as determined in Item (*2*), should not exceed 0.003 units.

Infrared Cells

23. (*a*) *General Considerations.*—The three most common types of infrared cells—demountable liquid, sealed liquid, and gas—will be considered under the following test procedures. Most of the window materials used for these cells require special handling techniques, particularly those that have a poor resistance to water (Table V). No attempt will be made to discuss each type of cell window in detail. Such information, including excellent descriptions of cells and methods of cell assembly, can be found in the literature (**9,10,12,13**).

Much of what was said regarding the general considerations of ultraviolet, visible, and near-infrared cells also applies to those used in the infrared region. For example, cells should be properly rinsed immediately before and after using, care should be taken to avoid touching the optical windows, and the cell should be wiped clean before placing in the cell holder. For infrared work it is not necessary that the cells be optically clear; however, they should be free of foreign matter. Contaminants can generally be detected by simply obtaining an infrared spectrum of the empty cell and noting the presence of any extraneous absorption bands. (Do not confuse with interference fringes, as described in the following test methods.)

(*b*) *Measurement Procedures:*

(*1*) *Leakage:*

Demountable Liquid Cell.—The cell, having been filled with the material of interest, should exhibit no evidence of leakage at the completion of the infrared measurement; that is, the window aperture should be full of solution. This cell is not generally recommended for quantitative analysis.

Sealed Liquid Cell.—Fill the cell with chloroform, using a hypodermic syringe. Plug the exit and inlet holes with tetrafluoroethylene plastic (Teflon) stoppers. Let stand at room temperature for a 3-hr period. The cell should exhibit no loss of liquid.

Gas Cell.—Connect the cell to a manometer and to a vacuum system. Evacuate the cell and isolate it from the vacuum system by some suitable means; for example, a stopcock. The pressure inside the cell should be less than 1 mm of mercury, as measured on the manometer. Allow to stand for 1 hr. The cell should exhibit less than 1 mm of mercury rise in pressure on standing.

(*2*) *Path Length:*

Demountable Liquid Cell.—Measure the thickness of the spacer used with a micrometer or some other suitable device. It should be understood that this measure is only approximate (one significant figure) and where circumstances require a more accurate value of path length, a sealed liquid cell should be used.

Sealed Liquid Cell.—Place the clean, empty cell in the sample beam of an infrared spectrophotometer. Adjust the instrument to record approximately 80 per cent transmittance. All other instrument settings should be those used for general qualitative analysis. It is important that the instrument should meet the calibration specifications as given in Part II on Wavelength Accuracy. Scan a region of the spectrum that yields a series of clearly defined interference bands, similar to Fig. 6.

If poor fringes are observed (less than 2 per cent peak to peak amplitude) the cell should be reconstructed, taking care to improve the optical flatness and parallel alignment of the cell windows.

The optimum spectral region to scan depends on the thickness of the cell. Table VI will serve as a rough guide in deciding which region of the spectrum to run. In the case of single beam instruments that record atmospheric absorption bands and thereby limit the usable wavelength regions, the recommended ranges do not apply. Here, judgment must be used.

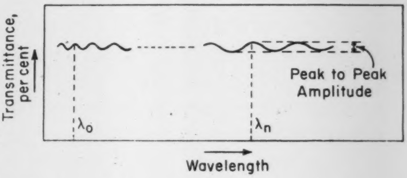

FIG. 6.—Typical Interference Pattern for a Sealed Liquid Infrared Cell.

TABLE VI.—SPECTRAL REGION.

Cell Thickness, mm	Wavelength, μ
0.0125	2 to 6
0.025	2 to 6
0.050	2 to 8
0.075	3 to 10
0.10	4 to 12
0.30	6 to 12
0.50	7 to 14

Calculate the thickness of the cell from the equation:

$$b = \frac{n}{2} \times \frac{\lambda_n \lambda_o}{\lambda_n - \lambda_o}$$

where:

b = thickness of the cell,

n = number of maxima between λ_o and λ_n ($n = o$ at λ_o), and

λ_o, λ_n = wavelengths of maxima (λ_n greater than λ_o).

or, if the instrument records on a linear wave number basis, then

$$b = \frac{n}{2(\nu_o - \nu_n)}$$

where:

ν_o, ν_n = wavenumbers of maxima.

Choose a value of n greater than 10.

NOTE 10: *Example.*—If $\lambda_o = 2.13\mu$, $\lambda_n = 6.95\mu$, and $n = 16$; then the thickness of the cell, b, is given by:

$$b = \frac{16}{2} \times \frac{6.95 \times 2.13}{6.95 - 2.13}\,\mu$$

$$b = 24.6\mu = 0.0246 \text{ mm}$$

Gas Cells.—Measure with a rule. Because of the relatively long path lengths, the percentage error will be inherently low.

(*3*) *Matched Cell Pairs.*—Choose a material that exhibits an absorption band or bands with an absorbance of about 0.4 to 0.6 at the cell thickness of interest (any wavelength region). Run a differential spectrum of the material in that wavelength region. The lack of compensation should not be greater than 1 per cent of the absorbance of the band or bands; that is, 0.004 to 0.006.

NOTE 11: *Example.*—Consider two 1.0-mm cells to be classed as a matched pair. Fill each of the cells with perchloroethylene (C_2Cl_4). Place one of the cells in the sample beam of the spectrophotometer and scan the region from 7.5 to 8.4 μ, using standard qualitative analysis instrument settings. Place the other cell in the reference beam and rescan the same region. The lack of compensation should not be greater than 0.006.

Cells which do not meet the above requirement may be used after determining the difference in absorbance between the cells using the appropriate reference solution in both cells at the wavelengths of interest and applying this difference as a correction factor to the absorbances measured in that cell.

REFERENCES

(1) K. S. Gibson, "Spectrophotometry," Nat. Bureau Stds., *Circular 484* (949).

(2) E. K. Plyler and C. W. Peters, "Wavelengths for Calibration of Prism Spectrometers," *Journal of Research*, Nat. Bureau Stds., Vol. 45, p. 462 (1950); RP 2159.

(3) Nicolo Acquista and E. K. Plyler, "Calibrating Wavelengths in the Region from 0.6 to 2.6 μ," *Journal of Research*, Nat. Bureau Stds., Vol. 49, p. 13 (1952); RP 2338.

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(5) E. K. Plyler, L. R. Blaine, and M. Nowak, "Reference Wavelengths for Calibrating Prism Spectrometers," *Journal of Research*, Nat. Bureau Stds., Vol. 58, p. 195 (1957); RP 2752.

(6) A. R. Downie, M. C. Magoon, T. Purcell, and B. Crawford, Jr., "The Calibration of Infrared Prism Spectrometers," *Journal*, Optical Society of America, Vol. 43, pp. 941–951 (1953).

(7) D. D. Tunnicliff, "Measurement of Nearby Stray Radiation in Ultraviolet Spectrophotometry," *Journal*, Optical Society of America, Vol. 45, p. 963 (1955).

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(10) "Synthetic Optical Crystals," Harshaw Chemical Co., Cleveland 6, Ohio.

(11) M. V. Buell and R. E. Hansen, "Spectrophotometry in the Far Ultraviolet," *Science*, Vol. 126, p. 842 (1957).

(12) "Infrared Sampling and Techniques," Instruction Manual, Perkin Elmer Corp., Norwalk, Conn., Vol. 11.

(13) R. C. Lord, R. S. McDonald, and F. A. Miller, "Notes on the Practice of Infrared Spectroscopy," *Journal*, Optical Society of America, Vol. 42, p. 149 (1952).

REPORT OF COMMITTEE F-1

ON

MATERIALS FOR ELECTRON TUBES AND SEMICONDUCTOR DEVICES*

Committee F-1 on Materials for Electron Tubes and Semiconductor Devices held three meetings during the year: in Skytop, Pa., on November 6, 7, and 8, 1957; in Washington, D. C., on February 26 and 27, 1958, and in Boston, Mass., on June 23 and 24, 1958.

At the Washington Meeting John R. Cuthill, of the National Bureau of Standards, spoke on "Field and Thermionic Emission Phenomena in Metals," and Frank J. Biondi, of Bell Telephone Laboratories, spoke on "Cleaning of Parts for Electron Devices."

The committee consists of 79 members, of whom 37 are classified as producers, 27 as consumers, and 15 as general interest members.

The officers elected for the ensuing term of two years are as follows:

Chairman, S. A. Standing.
Vice-Chairman, F. J. Biondi.
Secretary, S. Umbreit.
Assistant Secretary, C. L. Guettel.

A. P. Haase was appointed chairman of Subcommittee I, Cathode Materials, replacing A. M. Bounds, who resigned this position. E. A. Thurber was appointed chairman of Subcommittee VIII, Editorial, replacing C. L. Guettel.

The F-1 compilation of ASTM Standards on Electron Tube Materials has been published by the Society. This consists of the methods now carrying the F designation as well as other methods and specifications relating to the activities of the committee.

Liaison activities are continually maintained with other committees of the Society, including Committees E-2 on Emission Spectroscopy, E-3 on Chemical Analysis of Metals, and B-2 on Non-Ferrous Metals and Alloys.

RECOMMENDATIONS ACCEPTED BY THE ADMINISTRATIVE COMMITTEE ON STANDARDS

Subsequent to the 1957 Annual Meeting, Committee F-1 presented to the Society through the Administrative Committee on Standards the following recommendations:

Revision of Tentative Specifications for:

Tungsten Wire Under 20 Mils in Diameter (**F 288 - 54 T**),
Round Wire for Use as Grid Lateral Winding Wire in Electron Tube Grids (**F 290 - 54 T**), and
Nickel Alloy Cathode Sleeves for Electronic Devices (**F 239 - 57 T**).

The revisions of Tentative Specifications F 288 and F 290 were accepted by the Standards Committee on August 15, 1957, and the revision of Tentative Specification F 239 was accepted on December 13, 1957. The revised specifications appear in the 1957 Supplement to Book of ASTM Standards, Part 6.

The following new tentative methods were accepted by the Administrative Committee on Standards on March 31, 1958, and April 15, 1958, respectively, and these methods are appended hereto:[1]

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* Sixty-first Annual Meeting of the Society, June 22–27, 1958.

[1] The new tentatives appear in the 1958 Book of ASTM Standards, Part 2.

Tentative Method of Test for:

Volatile Content of Germanium Dioxide (**F 5 – 58 T**), and
Bulk Density of Germanium Dioxide (**F 6 – 58 T**).

NEW TENTATIVE

The committee recommends for publication as tentative the Specifications for Aluminum Oxide Powder as appended hereto.[1]

REAPPROVAL OF STANDARDS

The committee recommends the reapproval of the following standards which have stood for six or more years without revision:

Tentative Specifications for:

Round Nickel Wire for Lamps and Electronic Devices (**F 175 – 50**).

Methods of Test for:

Bend Testing of Wire (Wire for Radio Tubes and Incandescent Lamps (**F 113 – 41**),
Temper Strip and Sheet Metals for Electronic Devices (Spring-back Method) (**F 155 – 50**),
Testing Wire for Supports Used in Electronic Devices and Lamps (**F 157 – 50**),
Density of Fine Wire and Ribbon for Electronic Devices (**F 180 – 50**),
Strength of Welded Joints of Lead Wires for Electronic Devices and Lamps (**F 203 – 50**),
Surface Flaws in Tungsten Seal Rod and Wire (**F 204 – 50**),
Measuring Residual Stress in Cylindrical Metal-to-Glass Seals (**F 218 – 50**),
Testing Fine Round and Flat Wire for Electronic Devices (**F 219 – 50**), and
Measuring Mica Stampings Used in Electronic Devices and Incandescent Lamps (**F 652–51**).

TENTATIVES CONTINUED WITHOUT REVISION

The committee recommends the continuation without revision of the following tentatives:

Tentative Specifications for:

Round Chromium-Copper Wire for Electronic Devices (**F 268 – 56 T**), and
Molybdenum Wire Under 20 Mils in Diameter (**F 289 – 56 T**).

Methods of Test for:

Testing Sleeves and Tubing for Radio Tube Cathodes (**F 128 – 56 T**),
Sublimation Characteristics of Metallic Materials by Electrical Resistance (**F 278 – 55 T**),
Diameter by Weighing of Fine Wire Used in Electronic Devices and Lamps (**F 205 – 56 T**), and
Sag of Tungsten Wire (**F 269 – 52 T**).

Recommended Practice for:

Cathode Melt Prove-in Testing (**F 238 – 55 T**).

The recommendations in this report have been submitted to letter ballot of the committee, the results of which will be reported at the Annual Meeting.[2]

ACTIVITIES OF SUBCOMMITTEES

Subcommittee I on Cathode Materials (A. P. Haase, chairman) has set goals to be attained in the cathode materials field to include the measuring of cathode characteristics and predicting the life of the cathode; the ability to measure coating resistance and interface impedance separately; determining the photosensitive characteristics of cathodes; measuring the gas content of cathode materials; establishing good definitions for coating materials, including particle size, crystal structure, and reaction characteristics; further study of secondary emission in regard to magnetron type cathodes, and study of secondary emitter surfaces.

The *Electrical Test Section* continues work on the design of a super-clean standard planar diode, the task force on this project having now prepared a tentative specification describing this test device. Four companies have shown their intention of using the Standard Planar Diode by obtaining life racks.

The specification for the reference triode has been completed and is being reviewed editorially. It has been approved by letter ballot for recommendation as tentative.

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[2] The letter ballot vote on these recommendations was favorable; the results of the vote are on record at ASTM Headquarters.

New methods for measuring interface impedance are being studied, and investigations on cathode sublimation measurements are continuing. Exploratory work on cathodes operating under high voltage and pulse conditions is in progress, leading to a definite work program.

The *Physical and Mechanical Test Section* of this subcommittee continues round-robin tests on hot and cold collapse strength of cathode sleeves. Work is in progress on a revised tentative specification for disk cathodes.

Future work is planned on better measuring devices to give the accuracy required by the new tolerances in Specification F 239 – 57 T.

In the *Data Section*, consideration is being given to a new specification for statistically evaluating factory prove-in data of new melts of cathode material.

Consideration is being given to the possibility of factory testing of various heats of Cathaloy A-31. Testing on passive alloys may be an eventual interest of this section.

The *Cathode Coating Adherence Section*, recently formed from the task force investigating this subject, is preparing a bibliography of articles pertaining to cathode coating adherence.

The section is searching for a test for texture of applied coating and will attempt to define processing for standard devices in which materials can be compared and evaluated, the ultimate goal being a method of measuring adherence which would give numbers to permit the determination of different degrees of adherence.

Subcommittee II on Insulators (C. M. Harman, chairman) has made considerable progress on mica hole gage improvement. Two experimental model gages have been demonstrated, both appearing to have excellent possibilities. Further tests will be run and the models evaluated with respect to the mechanisms for controlling the lifting and seating of the tapered pin used to determine mica hole size.

Editorial changes have been made in Methods F 652 – 51. The proposed Tentative Specification for Aluminum Oxide Powder has been accepted for publication.

Subcommittee III on Strip (C. W. Horsting, chairman) has reviewed two standards for possible revision: Standard Method of Test for Temper of Strip and Sheet Metals for Electronic Devices (Spring-Back Method) (F 155 – 50), and Standard Method of Test for Density of Fine Wire and Ribbon for Electronic Devices (F 180 – 50).

Proposed tentative specifications for strip for radio tubes are nearing final approval.

Correlating results of the carbon adherence test with actual tube manufacturing performance continues.

Round-robin tests for gas content of strip have been conducted. The variations in results, seemingly due to differences in equipment and procedures, have stressed the need for a standard test method. Analysis of all test equipment and procedures will be made.

Subcommittee IV on Wire (D. R. Kerstetter, chairman) is balloting a proposed specification for round wire for use as grid side-rods in electron tubes. The subcommittee prepared the revisions of Specifications F 290 – 54 T referred to earlier. Also under consideration are revisions of Specification F 205 – 56 T.

The specification for miniature lead wire has been finally completed after consideration of the various tests for knot strength, and the specification has been submitted for letter ballot. Specification F 113 – 41 is being considered for revision. Further work continues in revising Specification F 219 – 50.

Subcommittee V on Metallic–Non-Metallic Seals (V. J. DeSantis, chairman).—The Task Group on Glass-to-Metal Seals has continued broad investigation to establish groundwork for the development and understanding of a means by which a glass-to-metal seal could be made and evaluated. A tentative specification for Kovar glass seal has been formulated. In conjunction with this, preliminary studies will be made on the use of other seals such as Dumet, tungsten, molybdenum, No. 52 nickel-iron alloy, No. 42 nickel-iron alloy, No. 4 alloy, and others.

Sources of information concerning the practices of sealing molybdenum and tungsten are being further investigated, with the ultimate aim of formulating a sealing and evaluation specification for this type of seal fabrication.

This group has also formulated a Tentative Method of Test for Iron-Cobalt-Nickel Alloy Glass Seals, using the simple "bead seal" and the "sandwich seal" for this purpose.

A Task Group on Standard Test Device for Metal-Ceramic Seals has conducted a round-robin test on ceramic test pieces, with sixteen companies participating. Items to be considered are: eccentricity; parallelism of the pulling faces; thickness of the metallizing and braze; type of metallizing (and maximum temperature); type of brazing alloy (and maximum temperature); load exerted during brazing; and breaking strength.

Other task groups are working on the specifications for Dumet, with considerable progress noted, and continuing the preparation of a specification for sealing glasses. A method of measuring the gas content of nickel wire when sealed into glass is under consideration by another task force.

Subcommittee VI on Semiconductors (H. C. Theuerer, chairman) prepared the Tentative Methods of Test for Bulk Density of Germanium Dioxide (F 6 – 58 T), and Tentative Methods of Test for Volatile Content of Germanium Dioxide (F 5 – 58 T) referred to earlier in the report. It is expected that publication of these proposed specifications will encourage further investigation and evaluation.

Subcommittee VII on Luminescent Materials (J. G. Koosman, chairman) has conducted extensive tests for wet and dry adhesion. The wet adhesion test will be drawn up as a recommended procedure. This test has included common sources of glass, potassium silicate, and barium acetate. A task group continues work on dry adhesion tests.

Interest in phosphor body-color measurement procedures is evident, and currently available techniques are being explored and resolved.

Three methods of measuring particle size of phosphors are being evaluated.

Subcommittee VIII, Editorial (E. A. Thurber, chairman) has reviewed six methods and specifications for other subcommittees.

Subcommittee IX on Materials Analyses (R. S. Kelly, chairman) has analyzed samples of the third lot of NiO, now designated as X673. Seven laboratories have participated in this analysis and are preparing statistical patterns of their findings. The problem of finding a pure and available base material is being explored.

A task group is working to establish methods for the determination of trace impurities in iron-nickel-cobalt glass-to-metal sealing alloy.

Work continues on analysis of clad materials. Close liaison with Committee E-3 has been maintained in this work.

Round-robin tests have been conducted on Cathaloy to test the efficacy of the pure nickel working curves when tungsten is present; final results have been reported. Methods for the determination of magnesium and tungsten in electronic nickel have been approved for reference to letter ballot.

Continued efforts will be made to set up a method for the vacuum fusion gas analysis of carbonized nickel and carbonized nickel clad steel.

This report has been submitted to letter ballot of the committee, which consists of 79 members; 41 members returned their ballots, of whom 37 have voted affirmatively and 0 negatively.

Respectfully submitted on behalf of the committee,

S. A. STANDING,
Chairman.

STANTON UMBREIT,
Secretary.

EDITORIAL NOTE

Subsequent to the Annual Meeting, Committee F-1 presented to the Society through the Administrative Committee on Standards the following recommendations:

New Tentative Method of Test for:

Electron Tube Materials Using Reference Triodes (F 8 – 58 T).

Revision of Tentative Method of Test for:

Diameter by Weighing of Fine Wire Used in Electronic Devices and Lamps (F 205 – 56 T), and

Sag of Tungsten Wire (F 269 – 52 T).

These recommendations were accepted by the Standards Committee on September 9, 1958, with the exception of F 8 which was accepted on November 13, 1958. The new and revised tentative methods are available as separate reprints.

REPORT OF COMMITTEE F-2

ON

FLEXIBLE BARRIER MATERIALS

The Board of Directors at its meeting of September 17, 1957, authorized the formation of a new technical committee, Committee F-2 on Flexible Barrier Materials. The organization meeting of Committee F-2 was held on December 12, 1957, in New York, N. Y.; the Steering Committee met on January 16, 1958, in Philadelphia, Pa.; and the committee and subcommittees met on May 28, 1958, in New York.

The scope for the committee has been defined as follows:

> *Scope.*—The development of definitions of terms and nomenclature, methods of test, and specifications for flexible barriers, including basic and composite materials and their application, and the promotion of research in this field. Standards covered by other committees shall be used when applicable.
>
> Typical flexible barrier materials are treated papers and fabrics, plastic films, and metallic foils, used alone or in various combinations.

The committee consists of 70 members, of whom 43 are classified as producers, 12 as consumers, and 15 as general interest members. Liaison contacts are maintained with other committees interested in flexible barrier materials.

The officers elected for the ensuing term of two years are as follows:

Chairman, C. C. Sutton.
Vice-Chairman, L. F. Swec.
Secretary, T. M. Hill.
Members-at-Large, W. B. Tibbets and James W. Goff.

ACTIVITIES OF SUBCOMMITTEES

Subcommittee II on Nomenclature and Definitions (R. B. Holmgren, chairman) has begun a literature search of all published definitions in the field of flexible barriers. This list will be circulated to the committee in order to determine which definitions should receive first priority.

Subcommittee III on Testing Methods (K. W. Ninneman, chairman).—In the area of transmission properties of flexible barrier materials, task groups were formed on Water Vapor Permeability (C. W. Desaulniers, chairman) and Gas Transmission (W. B. Kunz, chairman). Other transmission properties which will be eventually covered include odors and flavors, grease and oil, light, and absorption of water and chemicals.

An imposing list of mechanical or physical properties are being considered by the subcommittee. Of these, two will be studied by task groups as follows: Seal Strength (H. M. Weiner, chairman) and Tear, Impact Bursting, and Tensile Strength (C. P. Kulesza, chairman).

In the area of chemical properties, investigations are under way on chemical analyses, resistance to chemical reactions, biological characteristics, and corrosion inducing or inhibiting properties. A task group on Moisture Content (J. R. Hickey, chairman) was appointed.

Presently, the work of the subcommittee is largely that of a survey of published literature and industrial standards now in use. This published material will form the base of subcommittee work in the near future.

Subcommittee IV on Specifications (Paul Cope, chairman) will collect published data on the properties of flexible barrier materials. These data will serve to indicate those physical and chemical characteristics which should be included in flexible barrier specifications. These data will also show which test methods are needed to determine the properties within the specifications.

Respectfully submitted on behalf of the committee,

C. C. SUTTON,
Chairman.

T. M. HILL,
Secretary.

TECHNICAL PAPERS

With Discussions

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TENTATIVE FATIGUE STRENGTH REDUCTION FACTORS FOR SILICATE-TYPE INCLUSIONS IN HIGH-STRENGTH STEELS*

By H. N. Cummings[1], F. B. Stulen[1], and W. C. Schulte[1]

Synopsis

Values of the fatigue strength reduction factor for nonmalleable silicate inclusions in single-nucleus fractures of high-strength steel specimens are estimated. Data for the computations are taken from tests on 309 specimens of aircraft-quality SAE 4340 and 4350 steels, of 140, 190, 230, 260 and 300 ksi[2] ultimate tensile strengths (UTS). It is concluded that the values of the notch factors associated with these inclusions depend not only on the size of the inclusions but also on the hardness level of the steel. Although the sizes of the inclusions nucleating the fatigue failures ranged from only 0.001 in. to 0.003 in. in diameter, the fatigue strengths of those specimens having the larger inclusions were found to be substantially below those having small inclusions. Also, the reduction of fatigue strength by inclusions is more pronounced at the high hardness levels than at the 140 ksi ultimate tensile strength level. It is thought that for very small inclusions (less than 0.00025 in.) other inhomogeneities inherent in the steel itself may dominate the failure of a specimen.

In a previous paper on the Relation of Inclusions to the Fatigue Properties of High-Strength Steel (**1**),[3] the authors reported that practically all fatigue failures of specimens made from several heats of high-strength steels nucleated at small microscopic inclusions of the silicate type. It was determined, qualitatively at least, that the fatigue life and strength were dependent on the size of the inclusion at the origin of the crack if the stress was not too high above the fatigue limit. The present paper, which is an abbreviation of reference (**3**), deals with some studies of the quantitative aspects of the effect of nonmetallic nonmalleable inclusions on the fatigue strength of specimens of aircraft-quality SAE 4340 and 4350 steel in the tensile strength range from 140 to 300 ksi[2] ultimate tensile strength (UTS). These steels were produced by the basic electric-furnace process. The specific objective of the present study is the estimation of fatigue strength reduction factors for the inclusions that were the single-nucleus origins of fracture

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* Presented at the Sixty-first Annual Meeting of the Society, June 22–27, 1958.

[1] Consulting Engineer, Assistant Chief Engineer, and Chief Metallurgist, respectively, Curtiss-Wright Corp., Propeller Division, Caldwell, N. J.

[2] ksi = one thousand pounds per square inch.

[3] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 512.

of specimens tested in rotating-bending machines.

The data used herein include those used in reference (1) and a considerable amount of additional data reported in reference (2). These data are tabulated in reference (3). The 309 specimens from which inclusion size data are currently available came from tests of SAE 4340 steel of 140, 190, 230, and 260 ksi UTS and 4350 steel of 300 ksi UTS. The relationships obtained are discussed below.

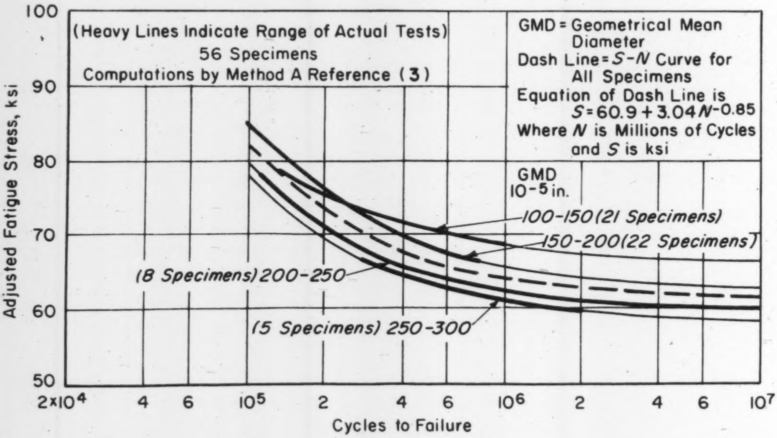

FIG. 1.—*S-N* Curves for Smooth Specimens Having Inclusions of Limited Sizes at Origin of Failure, 140 ksi UTS—SAE 4340 Steel.

TESTING DETAILS AND PROCEDURE

A full description of the testing procedures is given in reference (4). Briefly, the specimens, of curved planform, with minimum diameter of 0.230 in. and surface finishes of 2 to 5 microinches rms, were tested in R. R. Moore rotating-beam machines at 10,000 to 12,000 rpm. Practically all cracks leading to fracture originated at small microscopic inclusions of the silicate type that were located at, or very close to, the surface of the specimen. Fracture surfaces were examined at a magnification of approximately 400 ×, in polarized light, and the inclusions nucleating the failures were measured by use of a micrometer eyepiece.

The inclusions, which have already been observed to be more or less spheroidal (see photomicrographs, Figs. 37 to 41, of reference (4)) in these heats of steel, were measured parallel to and perpendicular to the surface of the specimen, and the geometric mean diameter (GMD), that is, the square root of the product of length and depth, was used as the characteristic size of the inclusion.

ANALYSIS OF DATA

In this investigation only those specimens having a single nucleus in the fractured surface were used in the analyses. It had been established that single-nucleus failures occur in a range of stress between the fatigue limit and an upper stress level that is about 30 per cent higher than the median fatigue limit. In this range the variance in stress was found to be substantially constant.

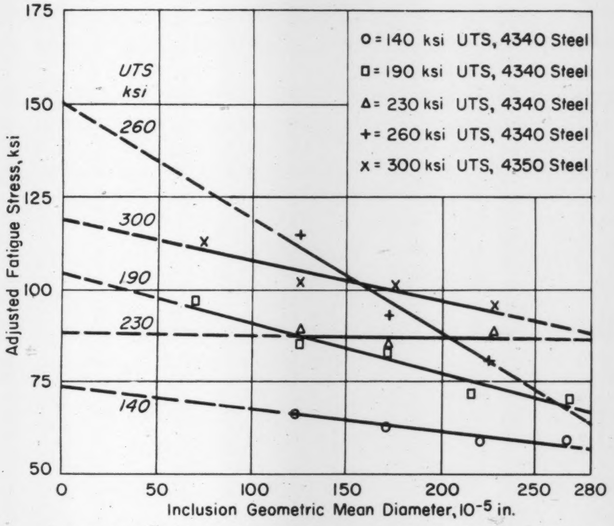

FIG. 2.—Average Fatigue Strength, for the Endurance Limit of Smooth Specimens Having Inclusions of Various Sizes at Origin of Failure.

Solid lines show range of actual tests—computations by Method A (**3**).

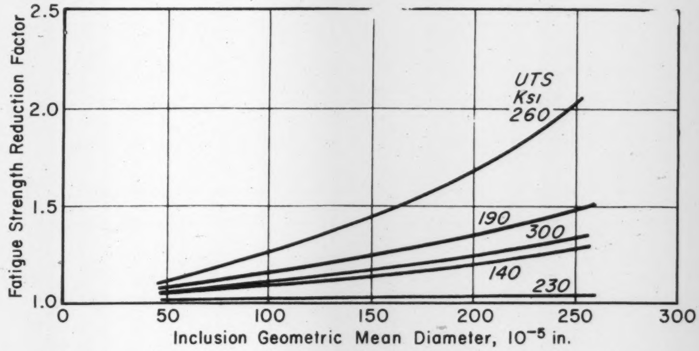

FIG. 3.—Tentative Fatigue Strength Reduction Factors of Nonmetallic Inclusions in SAE 4340 Steel and 4350 Steel, at the Endurance Limit.

Based on straight-line extrapolation, on Fig. 2, back to zero GMD. Computations by method A (**3**).

The general plan followed in trying to estimate stress concentration factors for the silicate inclusions found in the specimens studied was to divide the specimens into inclusion size groups. It was thought that if for each group an *S-N* curve could be obtained, it might be possible to extrapolate to an *S-N* curve for inclusion-free specimens. With such a curve available, ratios of strength with no inclusion to strength with inclusions of any size, that is, strength reduction factors, could be obtained. After curves such as those shown on Fig. 1 had been obtained (see reference (**3**), method A), it became apparent that extrapolation would give only approximate results. Therefore extrapolation was tried only at the "endurance limit" end of the curves, with results as shown on Figs. 2 and 3, giving computed values of strength reduction factors at the "endurance limit" only. (It should be pointed out that certain inconsistencies appear, on Figs. 2 and 3, in that the curves for the 230 ksi 4340 steel and the 300 ksi 4350 steel appear to be out of order with respect to the 140, 190 and 260 ksi 4340 steel. It is thought that since these two steels are relatively "brittle," the effect of the inclusions may be in some degree masked by some metallurgical factor related to brittleness.)

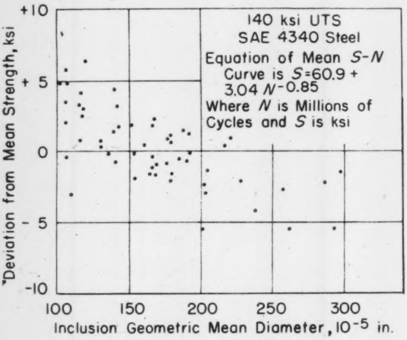

FIG. 4.—Deviations of Specimen Strength from Mean Strength *versus* GMD of Inclusions that Initiated Failure.

Computations by method B (**3**).

Next, as described in reference (**3**), method B, the effect of inclusions of each size-group in causing the strength of the group to deviate from the mean strength of all the specimens was determined. It was assumed that this deviation would be constant throughout the length of the S-N curve, and on that basis Figs. 4, 5, 6, and 7 were constructed, giving a second set of computed values of strength reduction factors at the "endurance limit" only. The factors thus obtained are somewhat smaller for the 140, 190, and 260 ksi UTS specimens of SAE 4340 steel and slightly larger for the 230 ksi UTS and the 300 ksi UTS 4350 steel than those obtained by method A.

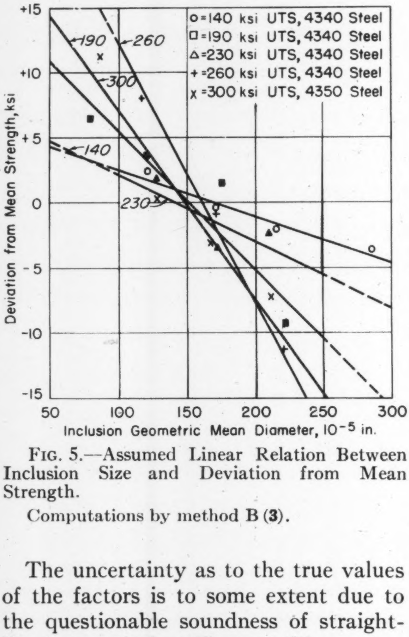

FIG. 5.—Assumed Linear Relation Between Inclusion Size and Deviation from Mean Strength.

Computations by method B (**3**).

The uncertainty as to the true values of the factors is to some extent due to the questionable soundness of straight-line extrapolation. To test this matter, the deviations determined under method B were separated into groups of equal numbers of specimens, as described in reference (**3**) under method C, and Fig. 8 was plotted for comparison with Fig. 5. According to Fig. 8, the relation between inclusion size and deviation of specimen strength from mean strength of all specimens is not linear. However, the eye-estimation curves drawn on Fig. 8 are not consistently curved either up

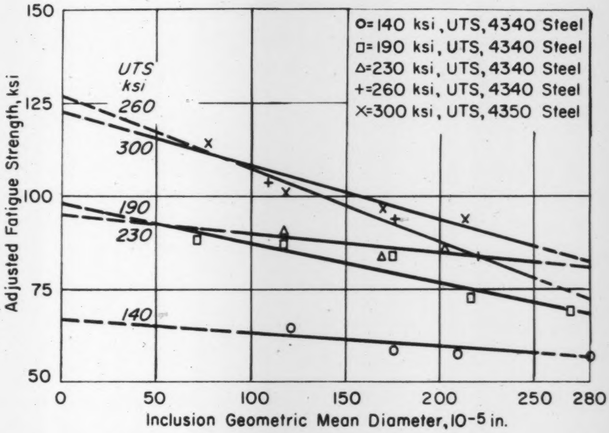

FIG. 6.—Average Fatigue Strength, for the Endurance Limit, of Smooth Specimens Having Inclusions of Various Sizes at Origin of Failure.

Solid lines show range of actual tests—computations by method B (**3**).

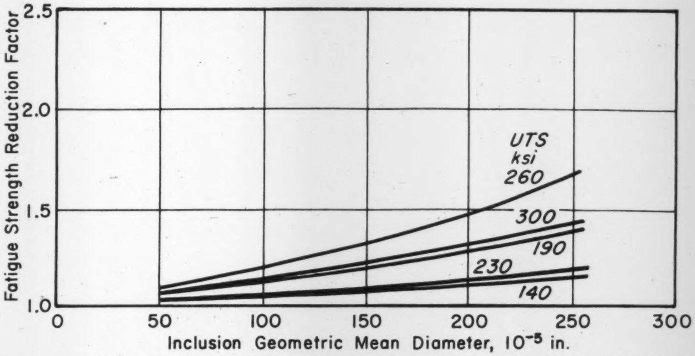

FIG. 7.—Tentative Fatigue Strength Reduction Factors of Nonmetallic Inclusions in SAE 4340 Steel and 4350 Steel, at the Endurance Limit.

Based on straight-line extrapolation, on Fig. 6, back to zero GMD—computations by method B (**3**).

or down. This could mean that the number of specimens is not nearly large enough to determine the true relationship. At least it is a clear warning that the quantitative results of this investigation are subject to considerable revision when an adequate amount of data become available. As a matter of fact, since reference (**3**) was prepared a considerable amount of additional data have become available. However, beyond determining that they will not greatly change the order of magnitude of the strength reduction factors, the authors have not yet had time to give them the thorough study that might produce answers to some of the questions that have arisen.

DISCUSSION

On examining Figs. 3 and 7, it is seen that the long-life fatigue strengths of these steels seem to depend not only on the inclusion size but also on the hardness level of the steel. It is important to note that neither of these trends can be explained by the mathematical theory of elasticity. First, the classical theory would not predict a change in the notch reduction factor (or stress concentration factor) when the hardness of the material is changed. Second, the stress concentration factor of a discontinuity, according to the classical theory, is not affected by the size of the discontinuity provided it is small in relation to the size of the cross-section. In other words, the notch reduction factor for a given type of inclusion should be constant regardless of its size and the hardness of the material.

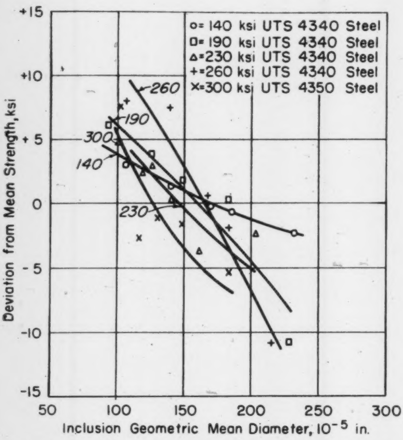

FIG. 8.—Assumed Nonlinear Relation Between Inclusion Size and Deviation from Mean Strength.

Computations by method C (**3**).

Several hypotheses may be advanced to explain these discrepancies. The effect of hardness on the sensitivity to an inclusion of a given size may be caused by the lack of sufficient ductility to allow yielding and consequent stress alleviation in the high stress region about the inclusion. Or it may be caused by residual tensile stresses around the inclusion, set up during cooling in the heat-treating operations.

Investigators in the past have observed the decrease in sensitivity of a discontinuity of a given shape when its size is decreased. It has been suggested that this decrease in sensitivity may be explained on the basis that the material behaves, in the limit, as though it were made of small blocks having a characteristic size. This characteristic size is usually associated with the grain size of the metal.

It is to be kept in mind that the conclusions of this report apply only to SAE 4340 and 4350 electric furnace steels so made that the dominant inclusions are the silicate type having more or less spheroidal shape. For example, other steel-making practices may lead to the stringer type of inclusions which probably have other notch reduction factors.

Another qualification on the results of the present studies must be pointed out. Recent studies of the mechanism of fatigue have revealed the complexity of the process—slip and reversed slip, accumulation of lattice vacancies, etc.—and have made it appear that the consumption of available fatigue life is occurring in many places in and near the region of maximum imposed stress rather than being entirely localized at the one point of maximum stress concentration. The present studies involve only "single-nucleus" fractures, and the inclusions listed in the tables, reference (**3**), were, for each specimen, the only ones visible at about 400× magnification. Placing responsibility for the fracture of a specimen entirely on the one visible stress raiser, that is, an inclusion, is a convenient fiction, but so far as the present report goes, there are not sufficient data to do otherwise.

The fatigue strength reduction factors shown in Figs. 3 and 7 indicate only the ratio of the fatigue strength, at the fatigue limit ($N = 10^7$), that the specimens might have, if there were no inclusions, to the strength the specimens actually had with inclusions of various sizes present. In other words, the factors are based on an assumed "inclusion-free" steel. Such inhomogeneities as grain boundaries, different orientations of adjacent crystals, different sizes of adjacent crystals, etc., cause different intensities of micro- and macro-stresses and consequently of strain and plastic deformation. The biasing effect of the normal forces on the slip planes—alternately tensile and compressive—cause microscopic permanent deformations to accumulate locally and to set up internal stresses that finally overcome the strength of the material and initiate one or more cracks. That is perhaps an oversimplified statement of the basic mechanism of fatigue, but it is sufficient for stating that probably the fatigue strength depends not only on the effect of inclusions but also on the simultaneous effect of smaller microscopic stress-raisers. The fact that of about 540 specimens of high-strength steels having single-nucleus fractures (reported in references (1) and (2)) six had no inclusions visible at 400× magnification and none had inclusions smaller than 0.00024 in. suggests that perhaps the relative effect of the inclusions decreases with decreasing size to such a degree that other inhomogeneities become dominant where inclusions are less than 0.00025 in. in size. If this be so, values read from Figs. 2 and 6 for fatigue strength at zero inclusion size may be overly optimistic. In other words, completely eliminating the silicate inclusions from the steel might not raise the fatigue strength as high as the straight-line extrapolations indicate.

CONCLUSIONS

From the data available for this analysis, the estimated trends suggest the following conclusions for aircraft-quality high-strength steels:

1. The medium and long life fatigue strength of the steels studied decreases substantially for each increase of 0.001 in. in the size of the silicate nonmalleable inclusions, particularly at the high hardness levels.

2. The fatigue strength reduction factor of an inclusion of given size generally increases as the hardness of the steel increases. At hardness levels below about 140 ksi UTS, inclusions of the sizes found are probably not serious stress raisers.

3. There is some reason to suspect that, in the steels studied in this investigation, any steel with no inclusions larger than about 0.00025 in. is in effect an "inclusion-free" steel, or at least a steel in which the effect of inclusions is insignificant. This suspicion is partly based on the fact that no inclusion smaller than 0.00024 in. geometric mean diameter (G.M.D.) has so far been found in the nucleus of single-origin fractures.

4. The mathematical theory of elasticity cannot account for the variations in the notch reduction factor of inclusions with inclusion size and hardness level of the steels. It is suggested that the sensitivity of inclusions at high hardness levels may be associated with high residual tensile stresses around the inclusions which are caused by differential thermal contraction during the heat treatment operations.

Acknowledgment:

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(**2**) H. N. Cummings, F. B. Stulen, and W. C. Schulte, "Investigation of Materials Fatigue Problems," Wright Air Development Center, *WADC TR 56-611*, March, 1957.

(**3**) H. N. Cummings, F. B. Stulen, and W. C. Schulte, "Fatigue Strength Reduction Factors for Inclusions in High Strength Steels," Wright Air Development Center, *WADC TR 57-589*, April, 1958.

(**4**) H. N. Cummings, F. B. Stulen, and W. C. Schulte, "Investigation of Materials Fatigue Problems Applicable to Propeller Design," Wright Air Development Center, *WADC TR 54-531*, May, 1955.

DISCUSSION

MR. R. E. KEITH.[1]—The authors' data presented in Fig. 2 may throw some light on a troublesome question of long standing in the literature—the shape of the curve relating endurance limit to static tensile strength for such steels as 4340. Mailänder,[2] for example, reports that the endurance limit increases continuously with increasing tensile strength, while Muvdi, Klier, and Sachs[3] show a more complex behavior of the curve in the range above 200,000 psi ultimate tensile strength, the curve having a negative slope between 200,000 and 220,000 psi.

While there are doubtless several variables which in combination account for these differing results, the present authors have demonstrated that inclusion size is an important variable. If the data in their Fig. 2 is crossplotted against tensile strength for a given inclusion size, one finds that for large inclusion sizes the curve obtained is reminiscent of Mailänder's endurance limit - ultimate tensile strength curve, whereas for small inclusion sizes the curve looks more like that of Muvdi, Klier and Sachs. (The use of two slightly different alloy compositions in the authors' work is of course an additional variable.) Since it is to be expected that the distributions of inclusions will differ in steels produced in different parts of the world using differing steelmaking practice, and since the authors have shown that inclusions exercise a considerable influence on fatigue strength of high-strength steels, one can more readily understand some of the varying results obtained by different investigators.

MR. F. B. STULEN (*author*).—The

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[1] Metallurgist, Research Laboratory, General Electric Co., Schenectady, N. Y.

[2] R. Mailänder, "Notch Sensitivity of Alloy and Carbon Steels Under Repeated Stress," *Stahl und Eisen*, Vol. 55, p. 39 (1935).

[3] B. B. Muvdi, E. P. Klier and G. Sachs, "Design Properties of High-Strength Steels in the Presence of Stress-Concentrations," Wright Air Development Center, *WADC Technical Report 55-103*, Supplement 1, June, 1955.

authors wish to thank Mr. Keith in pointing out that the size of inclusions present in different steels can readily account for the various shape curves reported in the literature when the fatigue limits of steels are plotted against the corresponding ultimate tensile strengths. A flattening or drooping of the curve of fatigue limit *versus* tensile strength, as the tensile strength is increased, is an indication of relatively large inclusions or other discontinuities while a continuously increasing curve probably indicates a clean steel relatively free of large inclusions. Perhaps the shape of this curve might be a good indication of the cleanliness of the steel.

MR. B. A. NIEMEIER, SR.[4]—I would like to ask the authors if they made any tests to determine the uniformity of the stress distribution in their specimen? To elucidate somewhat on this, it has been found, partly in our laboratory and other places, that our existing fatigue testing machines do not always provide the distribution which we would like to think actually exists. Someone has used the term parasitic stresses, to coin an expression, to define this.

Were there any tests to show that when two specimens of a like kind were put into a machine, there would be no machine variation or operator variation due to the assemblage of the specimen in the machine?

MR. STULEN.—As I mentioned in the presentation, we ran a large number of specimens distributed among ten machines. We then made a statistical analysis of these data and we found no significant difference among the various machines. This does not indicate that there is no variation within the machine itself. We estimate that there may be a variation of the order of 1000 psi in setting up the specimen in the machine. We have actually measured the vibratory load that occurs in the specimens due to eccentricities in rotation, and these were fairly small. We minimized these vibratory stresses by using a shock cord attached between the weight and the hanger. We made no special effort, however, to measure the stresses directly on the individual specimens.

MR. NIEMEIER.—The authors mentioned the residual stresses around the inclusions. These could be termed either micro or macro whichever is applicable, but was there any attempt to measure these residual stresses around the inclusions by some experimental technique?

MR. STULEN.—No attempt was made to measure the micro-stresses around the inclusions. It would be difficult to do this because the average inclusion is only 0.001 in. and the high stress region around the inclusion would be of one order of magnitude removed from this. The authors know of no experimental techniques for accomplishing these measurements.

MR. NIEMEIER.—I am inclined to agree that it is difficult. What we are attempting to do in some of our work is to study strain differences in small areas surrounding inclusions and large grains and get down to small sizes if we can. But I think Mr. Stulen has cleared these points for me sufficiently at this time.

MR. R. E. PETERSON.[5] (*by letter*).—The comments with respect to the stress concentrations at a hole near the edge of a plate are of interest. As the hole approaches the plate edge, the stress according to the theory of elasticity goes to infinity.[6,7] In fatigue, one would expect that the small section fails, and then the significant stress concentration becomes that of a "groove," whose bottom is the inside part of the hole. On this basis it can be shown that the stress concentration factor, K_f, of 3 for a hole in the middle of a very wide plate cannot in effect go beyond about 4.35 as the hole approaches the edge of the plate, and that this occurs when the small section is about one fourth the diameter of the hole. The effective increase of K_t is therefore limited to about 45 per cent.

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[4] Reynolds Metals Co., Structural and Mechanics Testing Laboratory, Richmond, Va.

[5] Manager, Mechanics Department, Westinghouse Research, Westinghouse Electric Corp., Pittsburgh, Pa.

[6] R. Mindlin, "Stress Distribution Around a Hole Near the Edge of a Plate in Tension," *Proceedings*, Soc. Experimental Stress Analysis, Vol. 5, No. 2, p. 614 (1948).

[7] R. E. Peterson, "Stress Concentration Design Factors," John Wiley & Sons, Inc., New York, N. Y., pp. 85, 107 (1953).

As a spherical cavity approaches the surface, the stress distribution is not known, but it is clear that it would be quite different from that of a cylindrical hole near the edge of a plate. I would like to ask the author (1) was any evidence of cracks of "break-through" type found? (2) was a higher K_f value obtained for specimens where the inclusions were a fraction of their diameter from the surface?

MR. STULEN.—Mr. Peterson had asked the authors whether the Neuber ρ' material constant had been estimated from the results of this paper. Although the Neuber ρ' value might be of interest in explaining the effect of the inclusion size on fatigue strength the authors were not able to estimate this factor since there were several variables required for this estimate that were not known.

For example, Jeffery[8] first showed that the stress concentration at a cylindrical hole in a plate increases rapidly as the hole closely approaches the edge of the plate. A rapid increase in the stress concentration occurs when the distance between the edge of the plate and the top of the hole becomes less than about one and a half radii. It is believed that this trend is also valid theoretically for any void or inclusion that approaches the surface of the metal. However, the theoretical stress concentration factors of a silicate sphere are unknown. It is for these and similar reasons that those quantities necessary to estimate the Neuber factor are unknown.

Concerning Mr. Peterson's question on the existence of non-propagating cracks of the "break-through" type, the authors' examination of the surfaces of the specimens was not accomplished at sufficient magnification to prove whether such cracks were present and failed to propagate. The lengths of these cracks if they exist, would probably be about equal to the inclusion diameter which, on the average, was slightly greater than 0.001 in.

There is considerable evidence given in the authors' previous work[9] that the K_f values of specimens having critical inclusions that are greater than one radius below the surface are considerable less than those associated with critical inclusions that are either open to the surface or are tangent to the surface. However, since there is a fairly large amount of scatter in these observations, it would be difficult to determine quantitatively the effect of the depth of the inclusion on the notch reduction factor.

MR. W. J. TRAPP.[10]—In respect to Neuber's theory, I would like to mention that the size of inclusions involved here is smaller than the elementary particle on which Neuber bases his theory and that his theory is not applicable in cases where radii are as small as we encounter in this case. I believe that the stress concentration here is mainly caused during quenching due to the difference in expansion coefficients of the two materials involved, namely, the aluminum oxide-silicate composition of the inclusions and the surrounding steel.

[See also discussion of paper by L. P. Tarasov, W. S. Hyler, and H. R. Letner, page 538.]

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[8] G. B. Jeffery, "Plane Stress and Plane Strain in Bipolar Coordinates," Philosphical, *Transactions*, Royal Soc., London, A, Vol. 221, p. 265 (1921).

[9] H. N. Cummings, F. B. Stulen and W. C. Schulte, "Investigation of Materials Fatigue Problems," Wright Air Development Center *WADC Technical Report 56-611*, March, 1957.

[10] Chief, Creep and Dynamics Section, Metals Branch Materials Laboratory, Wright Air Development Center, Wright-Patterson Air Force Base, Ohio.

TORSIONAL FATIGUE PROPERTIES OF SMALL DIAMETER HIGH-CARBON STEEL WIRE*

By Harry C. Burnett[1]

Synopsis

The torsional fatigue properties of small diameter high-carbon steel wire were investigated in three different types of test. In general, there was no direct correlation between these properties and the tensile strength of the wire. Comparison of a commercial cold-drawn wire with wire from a vacuum-melted heat showed that the torsional fatigue life of cold-drawn wire is decreased by the presence of inclusions. It was also found that oil-tempered wire stressed in torsion is less susceptible to the initiation of longitudinal shear cracks than cold-drawn music wire. Shot peening greatly increased the fatigue life of springs coiled from all three types of wire.

A machine for determining the fatigue properties of wire stressed in torsion at constant amplitudes of strain is described. Good agreement was found to exist between the results obtained with this machine and the results of fatigue tests on compression springs.

Small-diameter wire for springs is commonly specified on the basis of tensile strength. Previous work at the National Bureau of Standards and elsewhere had indicated that this was not an adequate criterion of the fatigue behavior of the wire under the torsional load to which it is subjected in helical extension or compression springs. It appeared, therefore, that additional information was needed on the metallurgical factors influencing the resistance of wire and springs to fluctuating load and on methods for evaluating this resistance. Accordingly, the torsion-fatigue properties of three different wires were determined under three different testing conditions. Also, as there is little published information on the effect of shot peening on the properties of music-wire springs, this factor was investigated on all three types of wire. These studies are part of a broad program sponsored by the U.S. Army Ordnance Corps, Springfield Armory.

Ramson (**1**),[2] Cummings (**2**), and others have shown that the fatigue strength of steels is affected by inclusions. Ramsom (**1**) states that the elimination of stringer type inclusions results in a large increase in the transverse fatigue limit so that it approaches the longitudinal limit. Previous work in this

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*Presented at the Sixty-first Annual Meeting of the Society, June 22–27, 1958.

[1] National Bureau of Standards, Washington, D. C.

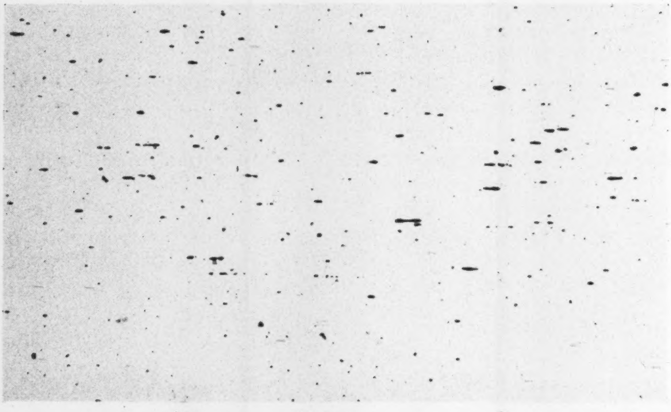
[2] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 525.

TABLE I.—CHEMICAL COMPOSITION (PER CENT BY WEIGHT) AND MECHANICAL PROPERTIES.

Wire	Carbon	Manganese	Phosphorus	Sulfur	Silicon	Tensile Strength, psi	Torsional Yield Strength[a], psi
AMA................	0.89	0.37	0.011	0.017	0.25	368 000	209 000
VMA................	0.88	0.47	0.001	0.004	0.27	350 000	196 000
AMA-OT...........	0.86	0.51	0.005	0.014	0.23	282 000	174 000

[a] The nominal surface shear stress corresponding to a surface shear strain offset of 0.0035.

(*a*) VMA.



(*b*) AMA.

FIG. 1.—Longitudinal Sections of 0.039 in. Diameter High-Carbon Spring Wires. Unetched (× 100).

laboratory (3,4) and elsewhere has shown that, in helical compression springs coiled from cold-drawn wire the initial fatigue cracks are parallel to the longitudinal axis of the wire and coincide with a plane of maximum shear stress. Stringer type inclusions in wire are drawn out parallel to the axis of the wire so that if such inclusions were eliminated the fatigue properties of springs coiled from the wire would be improved. Accordingly a supply of wire drawn from a heat of vacuum-melted steel of similar composition was obtained for comparison with commercial cold-drawn wire and with oil-tempered wire.

The preparation of compression springs for fatigue testing is time consuming, as special care is required in coiling, grinding, and measuring the spring. A machine was, therefore, designed to determine the torsional fatigue properties of spring wire without the need of coiling the wire into springs. The results of tests with this machine are compared with those on compression springs.

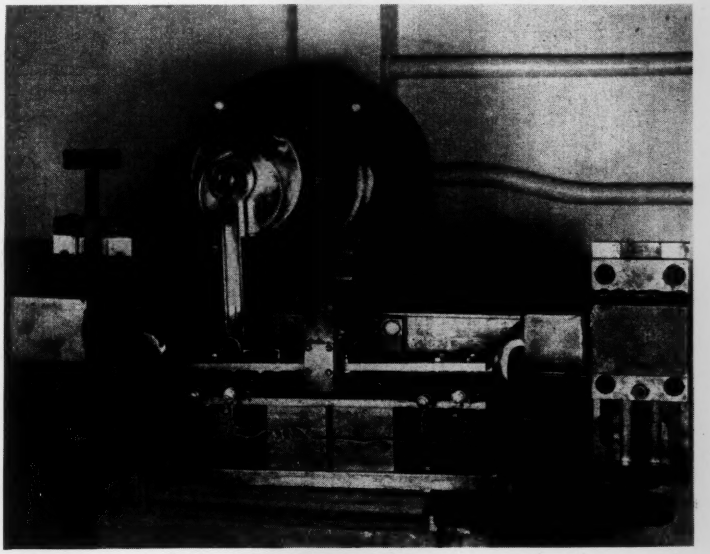


FIG. 2.—Fatigue Testing Machine for Short Compression Springs. Eight springs can be tested simultaneously.

MATERIAL AND TESTING PROCEDURES

The following three steels were used in this investigation: (1) commercial music spring wire, coded AMA, (2) commercial music wire which had subsequently been oil-tempered, coded AMA-OT, and (3) music wire which had been fabricated from a specially prepared billet of vacuum-melted steel, coded VMA. All wires were 0.039 in. in diameter. The chemical composition and tensile strength of the wires are given in Table I.

The AMA-OT wire had been solution treated at 1600 F, quenched in oil at 250 F, and tempered at 800 F.

Photomicrographs were made at 100

magnification of longitudinal sections of AMA and VMA wires. A comparison of the relative number and size of the inclusions may be made from Fig. 1. The VMA wire contained approximately one fifth as many inclusions as were found in the AMA wire.

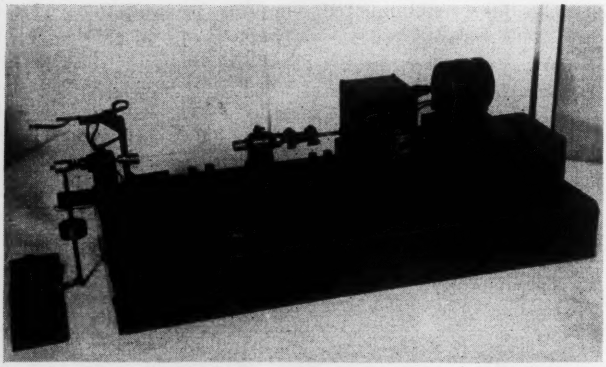


FIG. 3.—Machine for Fatigue Testing Wire in Reversed Torsion at Constant Maximum Torque.

Three types of fatigue tests were made on the wire as follows: (1) tests of short compression springs, (2) tests of straight wire under constant amplitude of torsional load and (3) tests under constant amplitude of torsional deflection. The spring tests were conducted in a "wobble-plate" type of machine (Fig. 2) which has been described previously (3).

A reversed-torsion machine operating at a constant amplitude of load, designed and constructed at the National Bureau of Standards, is shown in Fig. 3. The wire being tested is twisted in one direction, by means of a motor and reduction gear box, until the adjustable weighted arm at the left is raised to the horizontal (maximum torque) position. A contact is then closed which operates an electronic relay to reverse the motor, and the wire is twisted in the opposite direction until the weighted arm is again horizontal. The cycle is then repeated.

Most of the reversed torsion tests of straight wire were conducted under constant amplitude of deflection in a machine designed and constructed at the Bureau of Standards. Figure 4 shows the testing machine with a sample of 0.039-in. diameter music wire, *A*, in position to be tested. The grips holding the wire oscillate through an angle of about 120 deg. The amplitude of deflection in the wire is set by an adjustable coupling, *B*, that shifts the phase relation between the two chucks. A small amount of longitudinal tension is provided by a

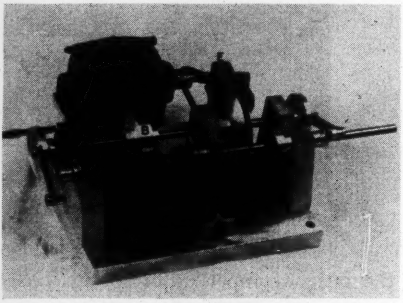


FIG. 4.—Machine for Testing Wire in Reversed Torsion at Constant Amplitude of Deflection.

(*A*) Specimen, (*B*) Adjustable Coupling, (*C*) Rubber Coupling, (*D*) Switch, (*E*) Reducing Gear Assembly and Counter.

rubber coupling, *C*, between the right front supports. When the specimen fractures, the right chuck is retracted by the rubber coupling, thereby operating a switch, *D*, to stop the machine. A counter is driven through a reducing gear assembly, *E*, attached directly to the motor shaft. Operating speed is approximately 350 cpm.

Cold-drawn wire has a much greater tendency to develop longitudinal fatigue cracks than oil-tempered wire; consequently, it is difficult to compare the two types of wire. In either the springs or the straight wire tested under constant amplitude of deflection, actual fracture may not occur until long after the formation of the initial crack in the cold-drawn wire (4). Accordingly the data are recorded in terms of the number of cycles to the start of cracking, as this provides the best measure of the useful life of springs.

Fatigue cracks in the springs were detected by periodic measurement of the deflection under static load. Presence of a crack resulted in decreased stiffness of the spring, which was readily detected in this way. During wire fatigue tests on the constant-deflection amplitude machine, the start of longitudinal cracking was detected by a device that was

TABLE II.—RESULTS OF REVERSED TORSION TESTS OF 0.039-IN. DIAMETER MUSIC WIRE, AMA 0.89 CARBON COLD-DRAWN WIRE.
Stress Amplitude 171,000 psi.

Condition	As Drawn			Straightened		
	Number of Tests	Average Cycles to Failure	Range of Results	Number of Tests	Average Cycles to Failure	Range of Results
As received	10	33	19	7	34	15
Shot peened	8	37	12	7	42	17
Stress relieved, shot peened	10	82	17	7	84	15
Stress relieved, shot peened, stress relieved	4	90	13	4	92	11

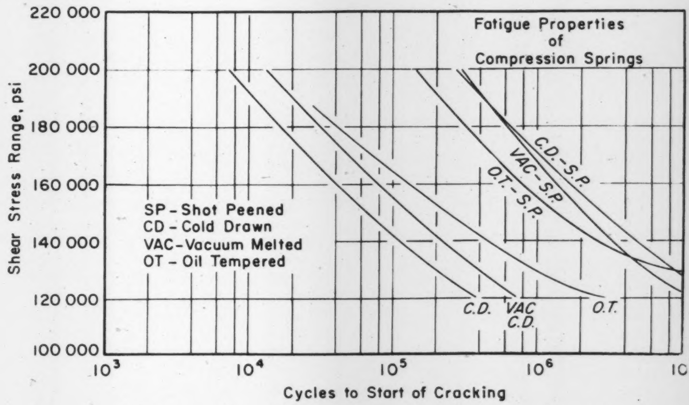


FIG. 5.—Results of Fatigue Tests on Springs Coiled from Cold-Drawn Music Wire, Oil-Tempered Wire, and Wire Cold-Drawn from Vacuum Melted High-Carbon Steel. Included Are the Results of Tests on Similar Springs in the Shot Peened Condition.

sensitive to the decrease in torsional stiffness of the specimen.

A Perkins precision spring coiler was used to coil the springs used in this investigation. All springs were flat ground on the ends after coiling and were of the following approximate dimensions: free height, 1.6 in.; spring index (mean coil diameter to wire diameter), 10; active coils, 4; inactive coils, 3 on each end. The shot-peening conditions for both wire and springs consisted of $\frac{1}{2}$ hr treatment with 0.009-in. diameter shot. The shot-peened springs were stress relieved at 450 F for 30 min and the remaining springs were stress relieved at 365 for 25 min. All springs were preset prior to testing by compressing to produce a nominal shear stress in the wire equivalent to approximately 75 per cent of the tensile strength. The initial preload on the springs when placed in the machine ready to be tested was approximately 15,000 psi.

As the main interest centered on the fatigue properties of springs in the high stress region, only the finite portion of the *S-N* curves are shown in this report.

RESULTS AND DISCUSSION

The effects of various combinations of stress relieving and shot peening were studied by testing samples of wire in the following conditions:

1(*a*) As drawn,
1(*b*) Straightened,
2(*a*) As drawn, shot peened,
2(*b*) Straightened, shot peened,
3(*a*) As drawn, stress relieved, shot peened,
3(*b*) Straightened, stress relieved, shot peened,
4(*a*) As-drawn, stress relieved, shot peened, stress relieved, and
4(*b*) Straightened, stress relieved, shot peened, stress relieved.

These tests were made in the constant load-amplitude machine with the mean stress zero and a maximum torsional stress amplitude[3] of 171,000 psi. As the cyclic speed of the machine is in the order of 1 cpm, a high stress amplitude was selected in order to decrease the time of the tests. The results are given in Table II. The fatigue life was longest for those samples that had been stress-relieved, shot-peened, and stress-relieved (condition 4), however, only slightly longer than for those samples that had been stress-relieved and shot-peened (condition 3). The torsional fatigue life of those samples that had been shot-peened, but without prior or subsequent stress relieving, was found to be only slightly greater than the "as received"

TABLE III.—RESULTS OF FATIGUE TESTS ON SPRINGS COILED FROM 0.039-IN. DIAMETER, 0.86 CARBON OIL-TEMPERED WIRE, AMA-OT).

(Prestress approximately 15000 psi)

Nominal Shear Stress Range, psi	Cycles to Failure
179 000	72 × 10³
172 000	63
168 000	31
167 000	77
166 000	77
164 000	75
159 000	91
157 000	120
153 000	160
153 000	370
151 000	127
150 000	84
148 000	868
146 000	457
143 000	188
143 000	2 575
142 000	1 241
140 000	2 116
126 000	9 486
125 000	3 340
125 000	2 014
125 000	10 828[a]
123 000	274
120 000	1 041
115 000	6 173
112 000	12 642[a]
106 000	10 008[a]

[a] Specimen did not fail.

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[3] Maximum torsional shear stress amplitude refers to the maximum shear stress in the outer fibers when stressed in one direction.

samples. Similar results were obtained by Lessells and Murray (5) using a Baby Rayflex machine to test 0.162-in. diameter, SAE-1065 wire.

TABLE IV.—RESULTS OF FATIGUE TESTS OF SHOT-PEENED SPRINGS, AMA 0.89 CARBON COLD-DRAWN WIRE.

Nominal Shear Stress Range, psi	Cycles to Start of Cracking
196 000	248 × 10^3
191 000	422
182 000	1 021
181 000	315
177 000	333
176 000	973
174 000	1 644
170 000	1 358
170 000	1 304
164 000	1 045
162 000	1 284
160 000	2 620
159 000	6 276
158 000	3 905
157 000	3 998
155 000	450
152 000	3 092
152 000	2 923
151 000	5 248
148 000	867
144 000	4 068
143 000	2 124
142 000	1 735
141 000	7 771
140 000	7 160
140 000	10 000[a]
136 000	10 000[a]
135 000	496
133 000	10 860[a]
130 000	496
130 000	10 000[a]
127 000	1 351
125 000	7 336
123 000	834
123 000	10 320[a]
121 000	10 342[a]
121 000	10 100[a]
120 000	7 432
119 000	10 860[a]
118 000	7 724
115 000	518
112 000	11 291[a]
108 000	11 291[a]

[a] Specimen did not fail.

The results of fatigue tests on compression springs coiled from the 3 types of wire are shown in Fig. 5 together with results from similar springs that had been shot-peened. Because of the large number of data, only the trend lines for 50 per cent probability of failure are shown. Tables III and IV are typical examples of the data obtained in this investigation.

The *S-N* curve for the springs coiled from the vacuum melted cold-drawn steel wire indicates an improvement in fatigue life over the commercial wire by a factor of 2 at a nominal shear stress range of 140,000 psi. As the primary difference in these materials is in the number and size of inclusions and stringers, it appears that the difference in fatigue life is due to the improved cleanliness of the vacuum melted material.

The *S-N* curve for springs coiled from music wire that had been subsequently oil-tempered indicates a still greater increase in fatigue life. At a nominal shear stress range of 140,000 psi the fatigue life is approximately 5 × 10^5 cycles or an increase by a factor of 5 over commercial (AMA) music spring wire. The principal difference between the music wire and the oil-tempered wire exists in their metallurgical structure (not shown). The music wire possesses a highly cold worked structure in which the grains have been fragmented and elongated along the axis of the wire, providing longitudinal crystallographic planes that inherently weaken the specimen in fatigue. When the wire is stressed in torsion, these planes of weakness lie parallel to a plane of maximum shear stress and afford means for the easy propagation of longitudinal shear cracks. If nonmetallic stringers are also present, the probable nucleation of this type of fatigue crack is increased. The oil-tempered wire has relatively uniform grains of tempered martensite and as recrystallization has taken place the inherent planes of weakness have been removed. These grains are more resistant to fatigue stressing and thereby increase the useful life of the springs.

The results of fatigue tests on shot-peened springs are included in Fig. 5. Comparison of the curves for the cold-drawn or oil-tempered springs indicates a significant increase in useful life due to shot peening. The scatter in the fatigue test results was also increased for the shot-peened springs. This is shown for the VMA springs in Fig. 6 where the bands represent an empirically determined approximation to the inter-quartile range of the data; that is, the bands enclose 50 per cent of the experimental points. These bands are similar for the other wires and indicate that the increase in fatigue life due to shot peening is somewhat counteracted by the increase in dispersion of the results at low stress levels.

Numerous investigators have attempted to explain the improvement of fatigue life by shot peening as due to either the effect of compressive stresses or the effect of cold working. While residual compressive stresses in the surface layers may be beneficial with respect to specimens stressed in bending, it is difficult to show how stresses of this type could possibly be beneficial to specimens stressed in torsion, especially when viewed in the light of the maximum shear stress theory of failure. Consideration of the above leads to the conclusion that the main contributing factors that tend to increase the fatigue life of shot-peened specimens stressed in torsion are the resulting highly cold-worked surface layers and the multiplicity of round bottomed stress raisers. These dents along the surface of the wire tend to proportion the applied stress and thus reduce the effect of any single stress raiser that may have been present due to die marks or imperfections formed during the fabrication of the wire.

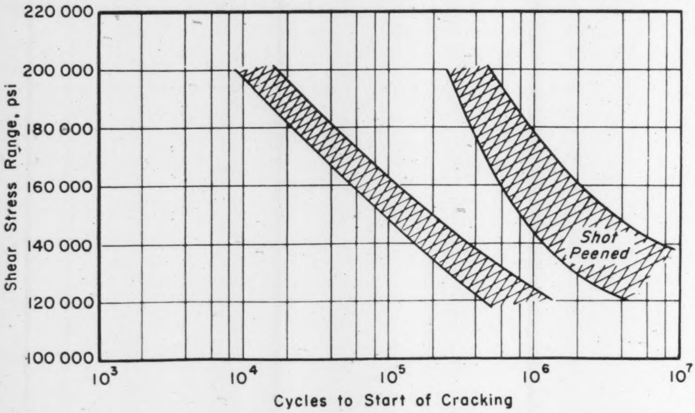

FIG. 6.—Results of Fatigue Tests on Springs Wound from VMA Cold-Drawn Wire and Similar Springs in the Shot-Peened Condition.

The cross-hatched bands indicate the approximate inter-quartile range of the data.

Samples of cold-drawn and oil-tempered wire were tested in reversed torsion at constant amplitudes of deflection. The results of these tests are given in Tables V and VI and are shown in Fig. 7. The *S-N* curves for compression springs wound from the same type of wires are included in Fig. 7 for comparison with the results of the reversed torsion tests. Good agreement was found to exist between the results obtained from the two methods of testing. The slightly better performance of the springs may be ascribed to the beneficial residual stress set up in the springs by the presetting operation. The residual stress, in this case, is a favorable body stress opposite in direction to the load applied during the test and varying from zero at the center of the wire to a maximum at the surface. The results, which are typical of those obtained in tests of several other wires, show that the reversed torsion test on straight wire provides a satisfactory indication of the performance to be expected of springs wound from the wire.

TABLE V.—AMA 0.039 IN. IN DIAMETER COLD-DRAWN WIRE TESTED IN REVERSED TORSION AT CONSTANT DEFLECTION AMPLITUDE.

Angular Twist per in., deg	Nominal Shear Stress Range, psi	Cycle to Start of Cracking
84	330 000	466
80	314 000	637
80	314 000	417
78	306 000	564
76	298 000	613
72	282 000	1 348
72	282 000	710
72	282 000	1 617
71	279 000	1 837
69	270 000	1 102
63	247 000	1 764
60	235 000	3 724
58	228 000	3 920
56	220 000	1 372
55	216 000	3 283
53	209 000	3 577
52	204 000	4 753
50	196 000	3 577
47	185 000	9 922
47	185 000	7 938
46	181 000	7 962
45	177 000	12 078
45	177 000	14 700
45	177 000	11 270
43	170 000	10 780[a]
41	161 000	20 188
40	157 000	22 540
37	146 000	24 010
37	146 000	30 502
35	138 000	34 912
34	134 000	38 318
34	134 000	23 275

[a] Broke in the grip.

The inadequacy of specifying tensile strength as a basis for expected fatigue life of wire stressed in torsion may be noted by comparing the tensile strength and fatigue properties of the three types of wire tested. It is the opinion of the author that specifications derived from reversed torsion tests would be of significant value to engineers and designers and would present a more realistic and usable indication of the torsional fatigue properties of wire.

TABLE VI.—AMA-OT 0.039 IN. IN DIAMETER OIL-TEMPERED WIRE TESTED IN REVERSED TORSION AT CONSTANT DEFLECTION AMPLITUDE.

Angular Twist per in., deg	Nominal Shear Stress Range, psi	Cycles to Start of Cracking
85	334 000	1 519
81	319 000	1 225
81	319 000	1 715
81	319 000	1 715
80	315 000	1 617
80	315 000	1 911
79	311 000	1 102
65	256 000	3 920
65	256 000	4 189
64	252 000	4 189
64	252 000	3 258
64	252 000	3 626
64	252 000	3 430
63	248 000	3 552
58	228 000	3 062
57	224 000	7 891
57	224 000	4 287
57	224 000	5 635
57	224 000	8 183
56	220 000	6 100
56	220 000	6 737
56	220 000	14 700
55	216 000	6 051
55	216 000	9 114
37	146 000	58 751[a]
36	142 000	40 940[a]
34	134 000	155 085[a]
34	134 000	175 787

[a] Broke in the grip.

CONCLUSIONS

1. The torsional fatigue life of cold-drawn music wire is decreased by the presence of inclusions that are drawn out with the wire.

2. The presence of inherent planes of weakness that lie in the direction of drawing, in cold-drawn music wire tend to decrease the useful torsional fatigue life of the wire.

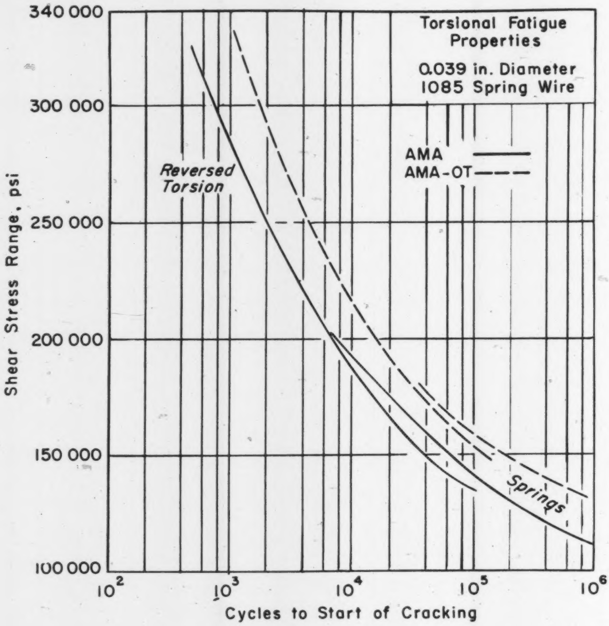

FIG. 7.—Results of Constant Deflection Amplitude Fatigue Tests of Wire and Fatigue Tests of Compression Springs Coiled from the Same Type Wire.

3. When stressed in torsion, oil-tempered 0.86 per cent carbon wire is less susceptible to the initiation of longitudinal shear cracks than cold-drawn music wire even though the tensile strength of the oil-tempered wire was the lowest of the three types of wire tested.

4. Shot peening greatly increases the reversed torsional fatigue strength of wire at high stress amplitudes. Shot peening accompanied by stress relief treatments significantly improves the pulsating fatigue strength of springs coiled from small diameter wire. Springs coiled from wire as small as 0.039 in. in diameter were successfully peened with 0.009 in. in diameter shot.

5. Shot peening followed by a stress relief at 450 F results in good fatigue properties and is recommended. Spring design must allow for adequate penetration of shot between adjacent coils to permit peening of the inner surfaces.

6. Good correlation exists between the data from fatigue tests (at constant amplitudes of deflection) on compression springs and on straight wire tested in reversed torsion at constant deflection.

Acknowledgment:

The author wishes to express his appreciation to J. A. Bennett, Chief of the Mechanical Metallurgy Section, National Bureau of Standards, for his constructive suggestions, to Houston Babb and D. B. Ballard for their assistance in conducting the tests and preparation of graphs and tables, and to the American Steel and Wire Division, U. S. Steel Corp. for their cooperation in the fabrication of the material used in this investigation.

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(3) H. C. Burnett and C. L. Staugaitis, "Endurance of Helical Springs Related to the Properties of the Wire," *Metal Progress*, Vol. 64, Sept., 1954, p. 77.

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DISCUSSION

MR. WILLIAM R. JOHNSON[1] (*presented in written form*).—The author's contributions to the basic knowledge of precision spring performance are always welcome. Springs in service and in testing almost always are subjected to unidirectional stressing at constant deflection. The net effect of any loads beyond the elastic limit is to cause plastic flow, thus lowering the applied stress and building up a favorable residual stress system. The elastic limit of spring materials in torsion is surprisingly low, when sensitive methods are used to measure it. A technique developed in our laboratories will pick up appreciable plastic flow in spring wires at stresses below 100,000 psi. Thus under most service conditions of springs some plastic flow may be expected. It may not be detected because the area of maximum stress is small.

Our test experience has been that particularly at high stresses the service life may increase if the yield point of the spring material is lowered by a high stress relief temperature. Of course, this is accomplished at the sacrifice of the ability of the spring to resist service loads without setting. With this as a preliminary, we notice that the materials the author has tested as springs represent three conditions of wire. His data for their torsional yield point rank them so that the lowest yield point material, the oil-tempered wire, has the best fatigue life, and the other two fall in line. The same conditions apply in reverse torsion at constant deflection. On this basis, we would question on the basis of the data available in this paper, the interpretation of the author's conclusions that the vacuum-melted music wire and oil-tempered microstructures are superior to conventional music wire. We suspect that much of the difference in results came from the difference in yield point. Does the author have comparative data on the three wires from his constant-load amplitude tester? What would be the results if the AMA wire were stress relieved at such a temperature that its yield point were the same as the AMA-OT wire? In our opinion, this additional information would add materially to the value of the author's work.

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[1] Assistant Director of Research, Associated Spring Corp., Bristol, Conn.

MR. H. C. BURNETT (*author*).—I thank Mr. Johnson for his comments and regret that I have no data on which to base an answer to the questions he has proposed.

MR. T. J. DOLAN.[2]—I am interested in the design details of the reversed torsion machine for gripping and testing individual strands of wire. It has been the experience of many people that with a design of this nature it is almost inherent that premature failure of the wire develops within the grips. I should like to inquire what was done to prevent failure from occurring prematurely due to the added unknown forces within the gripping devices of the machine which would, of course, invalidate the data as far as properties of the wire were concerned.

A second question is with respect to the curves in Fig. 5 which show only lines drawn in to represent the average final results. Is it possible that the scatter of the actual data if plotted on this graph would cover a band such that perhaps the three curves are within one normal population of scatter? Could the differences of fatigue life shown for, say, the cold-drawn *versus* the vacuum-melted or the oil-tempered wires be considered a reflection of the scatter from sampling in individual groups of tests? Were there really significant differences in these data when analysed on a statistical basis? The author's data indicate major emphasis on tests in the finite life region of the S-N curve; were any attempts made to actually determine the endurance limits of the wire or of any of the springs? In most instances for springs the fatigue limit would seem to be a more significant value than individual data on finite fatigue life.

MR. BURNETT.—In answer to Mr. Dolan's question about the grips, we did not experience any great difficulty. The way these grips were designed, the wire was bent at a right angle at each end. I have indicated in the paper the number of failures at the grips and it runs in the order of 2 to 3 in 50 or 60 wires. That might run 4 to 5 per cent; we did not feel that this presented a problem.

A comparison of the scatter bands for the three wires in the unshot-peened condition indicated there is a significant difference in the data. A similar comparison of the data for the wires in the shot-peened condition did not indicate a significant difference.

Fatigue tests on springs were carried out to 10^7 cycles. Emphasis was placed, however, on the finite portion of the curve. As stated earlier, these studies were conducted for the Springfield Armory in connection with ordnance applications where the design stress is frequently higher than the endurance limit. Infinite life is not a requirement.

MR. JOHN M. LESSELLS.[3]—The author concludes that oil-tempered 0.86 per cent carbon wire is less susceptible to the initiation of longitudinal shear cracks than cold-drawn music wire, even though the tensile strength of the oil-tempered wire was the lowest of the three types of wire tested.

The above conclusions are of interest and there is no intention here to challenge their validity. However, in view of the findings observed in the investigation of the failures of the Mt. Hope suspension bridge cables[4] it might be of general interest to refer to such. In this case the material was subjected to a bending stress as distinguished from a shearing stress, but it was possible to conclude from the paper and the resulting discussions that cold-drawn wire for such application was superior to the heat-treated grade.

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[2] Head, Department of Theoretical and Applied Mechanics, University of Illinois, Urbana, Ill.

[3] Lessells and Associates, Inc., Engineers, Boston, Mass.

[4] W. H. Swanger and G. F. Wohlgemuth, "Failure of Heat-Treated Wire in Cables of the Mt. Hope, R. I., Suspension Bridge," *Proceedings*, Am. Soc. Testing Mats., Vol. 36, Part II, pp. 2–84 (1936).

Mr. Burnett.—The question of the Mount Hope bridge always seems to come up whenever the use of oil-tempered wire is mentioned. Mr. Lessells' suggestion of making reference to Swanger and Wohlgemuth's paper on this subject is well taken, if only to help dispell some of the mistrust that seems to be connected with the use of oil-tempered wire. As Mr. Lessells has pointed out, the bridge wire failures occurred at areas subjected to bending stresses whereas springs are subjected to shearing stresses. Then too, considerable knowledge has been gained about oil tempering of wire since the time the wire was prepared for the Mount Hope bridge.

EFFECTS OF GRINDING DIRECTION AND OF ABRASIVE TUMBLING ON THE FATIGUE LIMIT OF HARDENED STEEL*

By L. P. Tarasov,[1] W. S. Hyler,[2] and H. R. Letner[3]

Synopsis

Although it has generally been assumed that parts ground transversely to the direction of applied stress would have a lower fatigue limit than those ground parallel to it, no such effect was found for two different grinding conditions.

In one instance, the residual grinding stress at the surface was compressive parallel to the length of the flat test bar for both grinding directions and the fatigue limit was the same for both groups of bars. In the other instance, involving good commercial grinding practice, this surface stress was tensile for longitudinal grinding but compressive for transverse grinding, and the fatigue limit was actually a little higher for the transversely ground bars than for the others. In both instances, the surface was strengthened sufficiently by means of compressive stresses in the longitudinal direction, as well as by cold work, to cause failures to nucleate below the surface and to raise the fatigue limit above the previously determined base-line value. The transverse grinding scratches did not reduce the fatigue limits, contrary to general expectations, because they could not exert a detrimental effect when the surface was strong enough to result in subsurface nucleation.

In a separate portion of this study, abrasive tumbling was found to raise the fatigue limit to a moderate extent, whether the surface had been previously stressed in tension or in compression by grinding. The tumbled surfaces were stressed very highly in compression and the failures nucleated below the surface.

The effects of various grinding conditions on the fatigue limit of hardened steel, in which the grinding direction was parallel to the length of the test bar and, hence, to the direction of applied stress, were reported in an earlier paper (**1**).[4] The principal conclusions of that study were:

1. Good commercial grinding practice did not affect the fatigue limit even though tensile grinding stresses of the order of 50,000 psi were present just below the surface.

2. Very severe grinding, which produced much higher tensile stresses, resulted in only a moderate reduction of the fatigue limit.

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[2] Battelle Memorial Institute, Columbus, Ohio.

[3] Lamp Division, General Electric Co., Cleveland Ohio; formerly Senior Fellow (Grinding Wheel Institute Fellowship), Mellon Institute of Industrial Research, Pittsburgh, Pa.

[4] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 538.

3. The substitution of a grinding oil for soluble-oil emulsion reduced or even eliminated the tensile stresses and introduced high compressive ones, and under some grinding conditions raised the fatigue limit considerably above the normal value.

Higher than normal fatigue values were associated with subsurface nucleation of the failures as opposed to the surface nucleation generally observed. It was not possible to explain all the results satisfactorily in terms of the residual grinding stresses alone, and it was necessary to bring in the cold working of the surface by grinding as an additional factor to be considered.

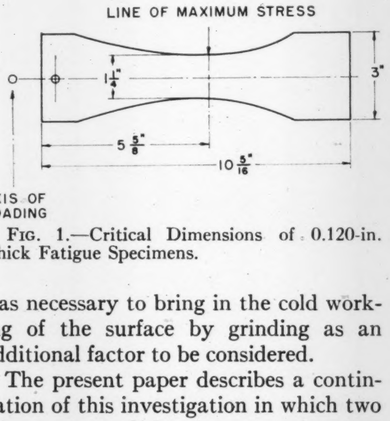

FIG. 1.—Critical Dimensions of 0.120-in. Thick Fatigue Specimens.

The present paper describes a continuation of this investigation in which two other aspects of abrasive processing were explored with respect to their effects on the residual stress and the fatigue limit. One had to do with the direction of the grinding scratches, the other with an entirely different type of abrasive operation, known as abrasive tumbling. In the scratch-direction study, the grinding conditions were kept the same, but the grinding direction was either parallel or perpendicular to the length of the test bar. The abrasive-tumbling study was designed to show the effect of this operation upon two kinds of bars: those that had been ground under good commercial conditions, which did not affect the fatigue limit; and those that had been specially ground with the aid of a grinding oil, so that their fatigue limit prior to tumbling was considerably higher than normal.

SPECIMEN PREPARATION

Preliminary Operations:

The flat fatigue bars were made from modified AISI 52100 type steel hardened to Rockwell hardness C 59, their shape and dimensions being shown in Fig. 1. The microstructure throughout the finished bars was a uniform dispersion of fine carbide particles in tempered martensite, and the only significant residual stresses were those introduced in the final processing. The details of the preliminary processing have been given in a previous paper (1), which also provided the residual-stress data and the fatigue limits for several longitudinal grinding conditions. The transversely ground and the abrasive-tumbled bars came from the same lot of steel as the longitudinally ground bars, and they were all processed in the same manner except for the final stage.

Final Grinding Operations:

The final grinding operations to a thickness of 0.120 in. were carried out on a horizontal-spindle reciprocating-table surface grinder with a white, vitrified bond, friable aluminum oxide wheel suitable for grinding hardened steels. The grinding conditions kept constant in this study were as follows:

Wheel: 38A46-I8VBE, 8 to 7 in. in diameter, $\frac{3}{4}$ in. wide;
Wheel speed: 6000 surface ft per min;
Table speed: 60 to 65 ft per min;
Unit crossfeed: 0.050 in. after each table traverse;
Unit downfeed: 0.001 in. after each complete crossfeed; and
Diamond dressing: moderately fast to leave wheel face open.

The grinding fluid was either a 1.25 per cent water emulsion of soluble oil or a sulfochlorinated grinding oil, denoted by *F* and *L* respectively in the previous paper, which gives the available information about their compositions.

The longitudinally ground bars were ground individually to their final thickness. The wheel was diamond dressed immediately prior to grinding each side so that the condition of the wheel face would be duplicated as nearly as possible during the last downfeed on both sides of all the bars comprising a group. These groups of bars were either left in the ground condition or else they were further processed by abrasive tumbling.

TABLE I.—RESIDUAL-STRESS AND FATIGUE DATA FOR GRINDING-DIRECTION STUDY.

	Group AG	Group BB	Group BC		Group BG	
Grinding fluid	Grinding Oil	Grinding Oil	Soluble Oil		Soluble Oil	
Grinding direction	Longitudinal	Transverse	Longitudinal		Transverse	
Roughness, microinches[a]	10	8	19		12	
RESIDUAL-STRESS DATA, PARALLEL TO GRINDING DIRECTION						
Method[b]	D	D	D	X_1	X_1	X_2
Surface stress,[c] psi	−87 000	−81 000	34 000	11 000	−24 000	−17 000
Peak tensile stress, psi	21 000	13 000	49 000	29 000	42 000	77 000
Depth of peak,[d] in.	0.0008	0.0007	0.0003	0.0004	0.0007	0.0003
RESIDUAL-STRESS DATA, PERPENDICULAR TO GRINDING DIRECTION						
Surface stress, psi	−150 000	−131 000	−37 000	−22 000	−60 000	−41 000
Peak tensile stress, psi	16 000	10 000	23 000	25 000	41 000	77 000
Depth of peak, in.	0.0008	0.0009	0.0008	0.0007	0.0007	0.0004
FATIGUE DATA						
Fatigue limit, psi	91 000	90 000	73 000		80 000	
Location of failure nucleation	Subsurface		Surface		Subsurface	

[a] Arithmetic average, measured perpendicular to grinding direction.

[b] Stress data in columns marked D are deflection-method averages for two surfaces; those in the X_1 columns are X-ray diffraction-method values obtained by Koistinen and Marburger for a single surface; and those in the X_2 column are X-ray diffraction-method averages obtained by Taylor for two surfaces.

[c] These are the stresses at a depth of 0.00005 in. below the ground surface. Positive and negative stresses are tensile and compressive, respectively.

[d] Depth below ground surface.

Transverse grinding had to be done somewhat differently since the bars projected beyond the edges of the 6-in. wide magnetic chuck. They were ground in three sections, the grip ends first and then the central 5-in. long section. It was possible to grind in such a manner that there was no measurable difference between the levels of the three sections, and all the fatigue failures occurred within the central portion.

In an attempt to simulate the wheel-face condition which existed during the last downfeed when the longitudinally ground bars were prepared, the bars were ground transversely in groups of four. The wheel was dressed after the second downfeed was completed on the center sections since this left approximately the same amount of material to be ground off from these portions of the four bars after dressing as from the whole surface of an individual longitudinally ground bar. Even with approximately equal amounts of stock removal after dressing, the condition of the wheel face during the last downfeed was not necessarily the same for the two grinding directions, since it is known that a wheel stays sharp longer when the cuts are interrupted than when they are continuous.

Groups AG and BC, for which the residual-stress and fatigue data are listed in Table I, were ground longitudinally with grinding oil and soluble oil, respectively, while groups BB and BG were ground transversely with the corresponding fluids.

Abrasive-Tumbling Operations:

Three groups of bars were abrasive tumbled after they had been longitudinally ground to a final thickness of 0.120 in. One of these groups, AH, was ground with a grinding oil just like the AG group bars, while the other two groups, BD and BF, were ground with a soluble oil in the same manner as the BC group bars. The data for these bars are presented in Table III.

The AH group bars were tumbled for 4 hr in an octagonal barrel of the triple-action type (with sloping sides) having a capacity of 10 cu ft. It rotated at 21 rpm and contained enough abrasive chips to half fill it. The water was level with the top of the load and contained about 0.4 oz of trisodium phosphate per gallon. The abrasive chips were fused aluminum oxide, No. 0 in size (maximum dimension in the range of 1 to 1½ in.). These tumbling conditions are representative of those used in practice to get the relatively rapid rate of cut needed for deburring and for appreciable rounding of sharp edges, except that a somewhat smaller chip size is generally used.

A few of the bars were found to be nicked after tumbling. It was thought that this resulted from occasional contact between bars, which were loose in the barrel. Therefore the other two groups of bars were clamped eight at a time in a fixture attached to the inside of a 6-cu ft octagonal straight-sided barrel, which was rotated at 8½ rpm in keeping with commercial practice. The bars were positioned with their lengths parallel to the barrel axis and with the flat surfaces tangent to a circle several inches from the barrel walls. They were tumbled for 4 hr but the direction of rotation was reversed at the end of 2 hr in order to equalize the action of the abrasive upon the long edges of the bars. The barrel was loaded three quarters full with chips and trisodium phosphate solution.

The BD group bars were tumbled with No. 0 chips, such as were used previously, the BF group bars with No. 4 chips, which are roughly ¼ in. in size. A number of the BD group bars were found to be nicked after tumbling, but none of the BF group bars were nicked. Evidently the nicks in the AH group bars, which were tumbled loose, were not due to occasional contact between the bars, as originally assumed, but to the use of chips that were too large and heavy for hardened steel bars having very sharp edges. Nicking did not occur when No. 2 chips, intermediate in size, were used for fixture-tumbling a group of bars that were not fatigue tested.

EXPERIMENTAL PROCEDURES

The deflection method used in the previous study provided the residual-stress data except for the BG group bars, ground transversely with soluble oil, which were analyzed by X-ray diffraction. The residual stresses were determined for both surfaces of one of these bars by A. A. Taylor of the Materials Development Laboratory of Pratt & Whitney Aircraft Division, United Aircraft Corp.; and for a single surface of another bar by D. P. Koistinen and R. E. Marburger of the Research Staff of General Motors Corp.[5] In both instances it was necessary to use specimens cut from the center portions of bars subjected to fatigue testing. It is assumed that the stresses were those present prior to fatigue testing since it has been shown that cyclic stressing did not measurably affect the residual stresses in similar bars (1). In addition, Koistinen and Marburger also obtained X-ray stress data for a specimen cut from the center portion of the same BC group bar that the authors had previously investigated by the deflection method. This bar had been ground longitudinally with soluble oil and had not been tested in fatigue. The X-ray data for the BC group bar, presented in Table I, showed good correlation with the deflection-method data obtained on a specimen from the same group of bars.

Fifteen bars in each group were tested in reversed cantilever bending, the fatigue limit being determined by the staircase method. The procedure and the method of evaluating the fatigue limit have been described in a previous paper (1).

EFFECT OF GRINDING DIRECTION

The experimental data are presented in Table I for the two groups ground with grinding oil (AG and BB), and with soluble oil (BC and BG). The surface roughness for the BC group is a little lower than previously reported. The indicated roughness value is the average for both surfaces of a number of bars instead of the average for a single bar.

With one exception, the residual stress curves were type 2, defined in the previous paper (1) as having a compressive stress at the surface which changed to a tensile peak just below the surface. The exception was the residual stress curve parallel to the grinding direction for the BC group bars. This curve was type 1, having a tensile stress at the surface that increased to a somewhat higher tensile peak.[6] The differences between the X-ray and deflection stress values for the same specimen in group BC are due in part to the fact that the former are for a single surface, while the latter are the average results for two surfaces that happened to differ considerably with respect to the residual stresses. The differences between the two X-ray determinations for group BG probably arise from real differences between the stresses present in two specimens as well as from differences in the X-ray techniques.

Grinding-Oil Results:

As is evident from Table I, the stress curves parallel to the grinding direction were practically the same for groups AG and BB. These had been ground longitudinally and transversely, respectively, with the aid of a grinding oil. This was likewise true of the stress curves perpendicular to the grinding direction, which were appreciably more compressive than those parallel to it. The peak tensile stresses were quite low and were essentially the same in both directions for each group.

In the direction parallel to the length of the bar (and to the applied alternating stress), the surface compressive stress was thus greater for transverse than for longitudinal grinding for the two groups processed with grinding oil. However, the fatigue limit was the same for both grinding directions so that the difference between the compressive stresses parallel to the length of the bar was not significant from the fatigue standpoint. For the good surface finishes that were obtained, the direction of the grinding scratches did not affect the fatigue limit because the surfaces were sufficiently strengthened by grinding to cause subsurface nucleation of failures.

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[5] In these X-ray analyses, Taylor used a special collimating-beam technique that he has developed, in which the slopes of the sides of the diffraction line determine the location of the peak. Koistinen and Marburger used another new technique, a description of which they plan to publish. This method involves the experimental conditions described by Christenson and Rowland (2) but with greatly simplified correction and calculation procedures, the peak positions being determined by a parabola-fitting technique.

[6] In Fig. 2 the curve for AG is type 2, and in Fig. 3 that for BC is type 1.

It should be pointed out that, just as in other cases of subsurface nucleation, the fatigue limit was appreciably higher than the base-line value previously established for the steel when the surface was neither strengthened nor weakened by grinding. The improvement in the fatigue limit associated with subsurface nucleation may be related to the large surface compressive stress parallel to the length of the bar. If this is the case, the larger compressive stress in the BB group bars was no more effective than the smaller one in the AG group bars. Another equally good possibility, based on previous results, is that the surface strengthening resulted from the cold work associated with grinding. Further research would be necessary to determine which of these factors is primarily responsible for subsurface nucleation.

Soluble-Oil Results:

The groups of bars ground longitudinally (group BC) and transversely (group BG) with soluble oil differed appreciably with respect to the residual stress curves for the two grinding directions, as shown in Table I. The grinding conditions for these groups were the same except for the grinding direction and the resulting change in the frequency with which the cut was interrupted. Parallel to the grinding direction, the stress was tensile both at and below the surface for the longitudinally ground bars, but it was compressive at the surface and tensile below for the transversely ground ones. The latter condition also applied to the stresses perpendicular to the grinding direction for both groups of bars. Thus the stress which is presumably effective from the fatigue standpoint, parallel to the length of the bar, was tensile at the surface for longitudinal grinding and compressive for transverse grinding, while the subsurface tensile peak was approximately the same for both grinding directions.

The fatigue limit for the longitudinally ground bars was essentially at the base-line value, which had previously been established as approximately 71,500 psi, and the failures nucleated at the surface. Changing the grinding direction to transverse raised the fatigue limit from 73,000 psi to 80,000 psi. The location at which failures nucleated also changed most significantly from surface to subsurface. Since subsurface nucleation implies that the surface is strengthened sufficiently to raise the fatigue limit above normal, it is concluded that the rather small increase in the fatigue limit is real.

It should be kept in mind that the observed increase in the fatigue limit for transverse grinding applies to the particular grinding conditions that were used. For other good grinding conditions or for other work sizes, there may be no improvement in the fatigue limit when the grinding direction is changed from longitudinal to transverse, but at least there is no evidence in the present limited study of the effect of grinding direction that the fatigue limit would be lower for transverse grinding as has been generally assumed.

Residual Stresses and Grinding Action:

The effects of grinding direction differed somewhat for the two types of grinding fluid used in preparing the bars. A comparison of the residual-stress data for hardened steel with those obtained by Halverstadt (3) for an iron-base high-temperature alloy (A-286) shows that the difference in the residual-stress pattern associated with the two grinding directions can also be affected by the work material. His specimens were rectangular in shape, 6 in. long by $\frac{3}{4}$ in. wide, so that the ratio of length to width was roughly the same as for the central portion of the hardened steel specimens. The residual-stress results for the two materials are listed in Table II, the values being rounded off for convenience. The data in the last two columns were obtained by X-ray diffraction by Koistinen and Marburger.

TABLE II.—RESIDUAL-STRESS DATA PARALLEL TO LENGTH OF BAR FOR LONGITUDINAL AND TRANSVERSE GRINDING.

	High-Temperature Alloy		Hardened Steel		Hardened Steel	
Grinding fluid	Grinding Oil		Grinding Oil		Soluble Oil	
Grinding direction	Longitudinal	Transverse	Longitudinal	Transverse	Longitudinal	Transverse
Surface stress, psi	−30 000	−30 000	−85 000	−130 000	10 000	−60 000
Peak tensile stress, psi	70 000	45 000	20 000	10 000	30 000	40 000
Depth of peak, in.	0.0007	0.0003	0.0008	0.0009	0.0004	0.0007

For the same grinding conditions in the two grinding directions, the surface stress was the same in one instance but much lower algebraically for transverse grinding in the other two instances. The peak tensile stress was either considerably lower for transverse grinding or else very nearly the same as for longitudinal grinding, the observed differences in the latter case being small enough to be accounted for by unavoidable variations between test bars. The depth of the tensile peak was smaller for transverse grinding in one instance, larger in another, and the same in the third instance. Thus for three sets of grinding conditions, the residual-stress curves for the two grinding directions differed quantitatively from each other in various ways, but their general configurations remained essentially the same.

EFFECT OF ABRASIVE TUMBLING

The residual-stress distributions parallel to the grinding direction (and to the length of the bar) are shown in Fig. 2 for a single surface of a representative bar from group AG, ground with a grinding oil, and for an AH group bar, ground in identical fashion and then tumbled loose for 4 hr with No. 0 fused aluminum oxide chips. The former is a type 2 curve (compressive stress at the surface changing to a tensile peak just below the surface) while the latter is type 3 (compressive stress at the surface which

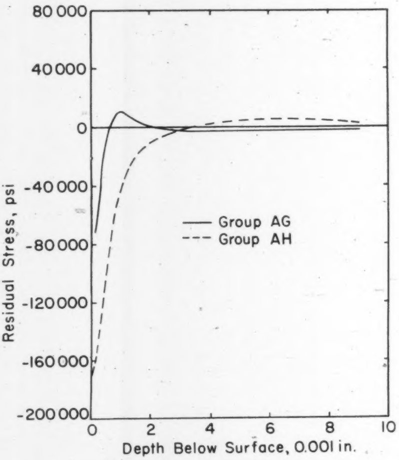

FIG. 2.—Typical Stress Distributions Parallel to Length of Bar.

AG, ground with grinding oil; AH, ground the same way and tumbled with No. 0 chips.

becomes very slightly tensile below the surface). Abrasive tumbling resulted in a considerable increase in the magnitude of the compressive stress and in the elimination of the small subsurface tensile peak.

TABLE III.—RESIDUAL-STRESS AND FATIGUE DATA FOR ABRASIVE TUMBLING STUDY.[a]

	Group AG	Group AH	Group BC	Group BD	Group BF
Grinding fluid	Grinding Oil	Grinding Oil	Soluble Oil	Soluble Oil	Soluble Oil
Type of tumbling	None	Loose	None	Fixture	
Size of tumbling chips	...	No. 0	...	No. 0	No. 4
Surface roughness, microinches	10	17	19	15	9
Residual-Stress Data—Parallel to Grinding Direction					
Surface stress, psi	−87 000	−168 000	34 000	−254 000	−270 000
Peak tensile stress, psi	21 000	0	49 000	0	6 000
Depth of peak, in.	0.0008	...	0.0003	...	0.0026
Fatigue Data					
Fatigue limit, psi	91 000	103 000	73 000	85 000	85 000
Location of fatigue nucleation	Subsurface		Surface	Subsurface	

[a] Footnotes *a*, *c*, and *d* of Table I are also applicable here; grinding direction was longitudinal and stress data are deflection-method averages for two surfaces except for BF group.

The average stress values for two surfaces from each group of bars, the fatigue limit and the surface roughness are presented in Table III. Although the fatigue limit for the ground bars was already considerably above the base-line value of 71,500 psi, abrasive tumbling raised it even further, from 91,000 psi to 103,000 psi. In both cases, the fatigue failures nucleated below the surface, showing that it had been appreciably strengthened by the processing.

Figure 3 and Table III show the residual-stress distributions obtained by grinding with soluble oil, group BC, or by fixture-tumbling with No. 0 or No. 4 aluminum oxide chips after grinding the same way, as in groups BD and BF. The latter curves are again type 3. Tumbling wiped out the tensile grinding stress at the surface and introduced an extremely high compressive stress, which was practically the same for both chip sizes. The subsurface tensile peak resulting from the grinding operation was also eliminated by tumbling with the larger chips, and only a trace of the peak stress remained when the smaller chips were used. Both tumbling operations increased

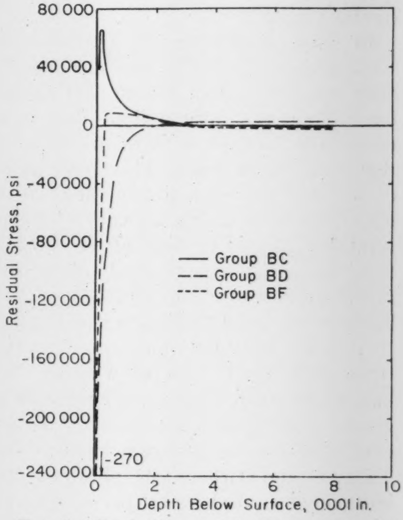

FIG. 3.—Typical Stress Distributions Parallel to Length of Bar.

BC, ground with soluble oil; BD and BF, ground the same way and tumbled with No. 0 or No. 4 chips.

the fatigue limit from 73,000 psi to 85,000 psi and changed the location of the failures from surface to subsurface.

Residual-stress curves were also obtained for bars processed like the BD and BF groups except that No. 2 abrasive chips, intermediate in size, were used. The curves were essentially the same as for the other two groups, tumbled with larger or smaller chips. Thus within the range investigated, chip size is likely to have little if any effect upon the improvement in the fatigue limit.

Although the surface stress prior to tumbling was tensile for the BD group bars and compressive for the AH group bars, tumbling with the No. 0 chips resulted in a considerably greater compressive stress for the BD group bars. Apparently the tumbling method affected the magnitude of the change since the BD group bars were tumbled in a fixture at a much lower rotational speed than the AH bars, which were tumbled loose.

In spite of the greater compressive stress present in the BD bars, the fatigue limit was lower than for the AH group bars. Thus the fatigue limit does not necessarily increase with an increase in the compressive stress. This is an indication that the microstresses associated with cold work, which can vary with the tumbling conditions, can affect the fatigue properties independently of the residual (or macroscopic) stresses, just as has been observed to happen in grinding.

Abrasive tumbling increased the fatigue limit by 12,000 psi whether the bars had been previously ground with a grinding oil or with a soluble oil. However, this is probably a coincidence since Tarasov and Grover (4) obtained an increase of around 30,000 psi as a result of tumbling bars that had been ground so severely as to reduce the fatigue limit far below the base-line value.

The surface roughness did not correlate at all with the fatigue limits. Tumbling increased the roughness of the bars ground with a grinding oil (from 10 to 17 microinches), and decreased it for those ground with soluble oil (from 19 to 15 or 9 microinches). In all three instances, the fatigue limit was raised, but it was highest for the AH group bars, which were the roughest of those that were tumbled.

As mentioned earlier, the very sharp edges of some of the AH and BD groups of bars became nicked during tumbling with the No. 0 chips. Some of these bars failed earlier than comparable bars that were not nicked, but some did not, so no definite conclusion can be drawn at present as to the significance of small nicks on the fatigue strength of abrasive-tumbled parts. Apparently the weakening effect of a nick viewed as a geometrical stress-raiser can in some instances be overcome by localized strengthening at the base of the nick. Regardless of this, nicking is undesirable and is always avoided in good tumbling practice.

Marble and Ruehrwein (5) have recently found that the fatigue limit for AISI 4130 steel hardened to Rockwell hardness C 35 increased appreciably with lower surface roughness. However, their results do not contradict those obtained in the present work since their wide range of roughness values resulted from changes in the processing method, which included such diverse ones as shaping, grinding, electrodischarge machining, and abrasive tumbling. The observed changes in the fatigue limit with the changes in the processing method and the resultant surface roughness were undoubtedly due to both factors and not just to roughness alone. Also, it is probable that many of their fatigue failures started at the surface, in which case the roughness would be expected to be a significant factor.

CONCLUSIONS

The conclusions of this study are derived from reversed-bending fatigue limits obtained for flat bars made from modified AISI 52100 steel uniformly hardened to Rockwell hardness C 59 and substantially free from residual stresses other than those introduced by grinding or abrasive tumbling.

Changing the grinding direction from longitudinal to transverse relative to the length of the bar either left the fatigue limit the same or else increased it by about 10 per cent. Even though the fatigue limit may be no higher for transverse than for longitudinal grinding under other grinding conditions, no evidence was found in the present study that it would be lower as has generally been assumed.

The fatigue limit remained the same when grinding oil was used in the preparation of the bars, and its value for longitudinal grinding was already considerably above the base-line value. For both grinding directions, the surface stresses were compressive and the failures nucleated below the surface.

The increase in the fatigue limit occurred when soluble oil was used under grinding conditions that were representative of good commercial practice. In this case, the fatigue limit for longitudinal grinding was at the base-line value. The surface stress changed from tensile to compressive when the grinding direction was made transverse instead of longitudinal, and the location of failures changed from surface to subsurface.

Abrasive tumbling of bars ground either with grinding oil or soluble oil resulted in very high compressive surface stresses. The failures all nucleated below the surface and the fatigue limits were raised 12,000 psi above those obtained for the corresponding ground conditions. The fatigue limits for the tumbled bars could not be correlated with the surface stresses. This indicated that the cold-worked condition of the surface can also affect the fatigue properties, just as has been found true in connection with grinding.

Acknowledgments:

The work reported in this paper was part of a joint program concerned with the effects of grinding and of abrasive tumbling on the residual stresses in hardened steel and on its fatigue properties. The bars were prepared at Norton Co. The residual-stress studies were carried out at the Mellon Institute of Industrial Research as part of a wider investigation of residual stresses sponsored by the Grinding Wheel Inst. and the Abrasive Grain Assn. The fatigue studies were carried out at Battelle Memorial Institute under Norton Co. sponsorship.

The authors wish to acknowledge the help of R. J. Favor in obtaining the fatigue data; and to thank A. A. Taylor, Pratt & Whitney Aircraft Division, United Aircraft Corp., and D. P. Koistinen and R. E. Marburger, General Motors Research Staff, for their assistance in providing some residual-stress data that were needed to complete the analysis of the results.

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DISCUSSION

MR. WILLIAM R. JOHNSON[1] (*presented in written form*).—We are particularly interested in the author's remarks on the effect of abrasive tumbling on the fatigue limit. Our test work in the precision spring industry is primarily concerned with the fatigue tests of specimens in one direction stressing at a constant deflection, and these remarks apply to such tests. Our tests on flat strips of pretempered spring steel are carried out on specimens mounted as cantilever beams with the mean stress equal to half the maximum stress. The test is insensitive to slight amounts of plastic flow in bending.

Somewhat to our surprise, several years ago we found that tumbling operations, which we thought of as quite mild, would increase the fatigue life markedly. For example, tumbling strips in sand moistened with oil, intended to be a cleaning operation, increased the endurance limit about 10 per cent or more, and the life at high stresses correspondingly. However, this gain was not pure profit. One of the important properties of a spring is its ability to resist high stresses elastically, without permanent set. These tumbled parts, which must have had considerable residual compressive stress in the surface layers, resulting from the tumbling, displayed a very much lowered elastic limit in bending. We measured this by checking the minimum diameter to which the flat strip could be deformed without taking set. The tumbled test strips, in other words, had a higher fatigue life and lower set point than the nontumbled parts. That this was due to the residual stresses was indicated by the fact that tumbled specimens treated at 600 F were restored to their former condition, with the fatigue life decreasing and set point increasing after the stress relief.

This has taught us to be suspicious of the "mild polishing so as not to change the surface" technique so often reported by investigators in the fatigue field.

MR. B. A. NIEMEIER.[2]—The authors have shown a number of curves on which residual stresses are plotted as a function of depth below the surface. I am curious as to the amount of scatter actually obtained with the technique which was employed. Perhaps they can indicate how closely that curve can be plotted and some of the experimental points from which these curves were drawn.

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[2] Reynolds Metal Co., Richmond, Va.

MR. L. P. TARASOV (*author*).—Mr. Johnson's experience that the fatigue limit can be raised appreciably by tumbling in sand extends greatly the range of particle sizes that can be used in abrasive tumbling operations to improve the fatigue properties. This, of course, is very helpful from the practical standpoint since numerous other considerations enter into the selection of the tumbling medium.

The drop in the elastic limit is an undesirable consequence not only of abrasive tumbling but also of other processes in which the surface is plastically deformed and compressively stressed, such as shot peening. This effect is important in small springs, but it can be readily overcome by stress-relieving, as shown by Mr. Johnson. In a larger spring, however, the affected layer is too thin in relation to the cross-section to have any adverse effect on the elastic behavior of the spring.

With regard to Mr. Niemeier's question about the scatter of the experimental stress data, there is practically none for the points upon which a single curve is based. This question was discussed in a previous paper[3] by one of the authors, where the curvature of a test specimen is plotted in Fig. 9 against the total thickness of the layer removed by etching. The deviation of the experimental points from the best smooth curve that can be drawn is barely perceptible. The residual stresses are calculated from these curves so that the very small experimental scatter in the curvature data is of no significance.

The accuracy with which a residual stress curve can be determined for an individual specimen is far greater than the reproducibility of the results obtained from a group of similarly processed test bars. This is especially true of very severe grinding conditions. In one such instance, the peak tensile stress was almost twice as high in one bar as in another, both ground under the same nominal conditions. Under good grinding conditions, the scatter is considerably less from bar to bar, but it is still greater than the accuracy with which a single stress curve can be determined.

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[3] H. R. Letner, "Residual Grinding Stresses in Hardened Steel," *Transactions*, Am. Soc. Mechanical Engrs., Vol. 77, pp. 1089–1098 (1955).

DISTRIBUTION OF FATIGUE FAILURES IN FLAT HARDENED STEEL TEST BARS*

BY W. S. HYLER,[1] L. P. TARASOV,[2] AND R. J. FAVOR[1]

SYNOPSIS

The locations of fatigue nuclei in flat hardened steel bars tested in cantilever bending were analyzed with respect to their distributions in the three principal directions. The surfaces of these bars had been prepared under various grinding and abrasive tumbling conditions. Analysis showed that the mean longitudinal fracture location was near the maximum stress section and that the location was relatively insensitive to grinding or tumbling conditions, relative stress level (ratio of applied stress to fatigue limit), and whether failure nucleated at or below the surface.

The transverse locations of the fatigue nuclei were distributed fairly uniformly along the flat surfaces of the bars with only slight evidence that the corners of the specimens were more frequent sites for fatigue nucleation.

Some grinding conditions and all tumbling conditions resulted in improved fatigue strength over a base fatigue limit. In these cases fractures nucleated predominantly subsurface, generally at inclusions.

A study was made of the kind, size, and depth of inclusions in those series of bars for which failure was predominantly subsurface. Of these bars, 93 per cent had inclusions at the origin; about 55 per cent of these were of the complex oxide type, while the rest were titanium cyanonitride inclusions. The mean diameter of the oxide inclusions was 0.0037 in.; the average depth was 0.0041 in. Corresponding values for the titanium cyanonitride inclusions were 0.0006 and 0.0011 in., respectively. No positive correlations were found between mean diameter and either relative stress level or fatigue lifetime, or between depth and either relative stress level or fatigue lifetime.

Two previous papers by Tarasov, Hyler, and Letner (**1**,**2**)[3] presented an evaluation of the effect of various grinding and abrasive tumbling conditions on the residual stress and fatigue limit of hardened steel. The observed fatigue limits varied over a wide range. No simple correlation was found between residual stress and fatigue limit, and the results suggested that surface cold work from grinding was an important factor.

Some additional studies were made of the location of the fatigue nuclei in the bars and of the type and size of inclusions that were frequently associated with the nuclei. This paper contains a summary and discussion of these studies.

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*Presented at the Sixty-first Annual Meeting of the Society, June 22-27, 1958

[1] Battelle Memorial Institute, Columbus, Ohio.

[2] Research and Development Department, Norton Co., Worcester, Mass.

[3] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 549.

The fatigue program had been aimed at studying as many grinding and tumbling variables as possible. Most of the specimens, therefore, were tested at or near the various fatigue limits, and few data were available for study from the finite lifetime region. Since many of the minor variations in processing would not be expected to influence fracture location, certain similarly processed and tested groups were combined to provide a larger population for each of the analyses that were made. There were four such groupings of processing and testing variables:

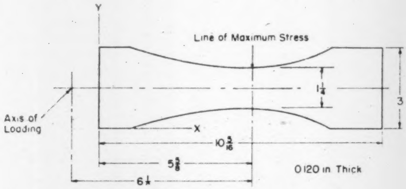

FIG. 1.—Fatigue Specimen Details.

1. Longitudinally ground bars tested in reversed bending,
2. Longitudinally ground bars tested in unidirectional bending,
3. Transversely ground bars tested in reversed bending, and
4. Abrasive tumbled bars tested in reversed bending.

DESCRIPTION OF SPECIMENS

The fatigue specimens were made from a modified AISI 52100 type steel, hardened to Rockwell hardness C 59. The microstructure throughout the bars was a uniform dispersion of fine carbide particles in tempered martensite. Figure 1 shows essential details of the finished specimen. The edges of the test section have elliptical contours with a minimum width of 1.25 in. The details of the processing have been discussed in the two previous papers (1,2) for each of the individual groups of fatigue bars.

The specimen, when clamped in the fatigue machine, was subjected either to a completely reversed or a unidirectional bending stress cycle. The specimen was bent as a cantilever and because of its configuration the nominal longitudinal stress, Mc/I, was not uniform along the test section. As indicated in Fig. 2, this stress was maximum at 6.5 in. from the loading axis. However, for an appreciable length, the stress was within 95 per cent of the maximum value.

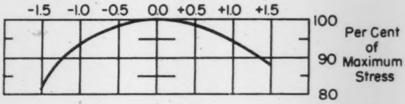

FIG. 2.—Longitudinal Stress Distribution.

The X and Y axes shown in Fig. 1 are the coordinate axes from which measurements were made of the longitudinal and transverse location of each fatigue failure. After each failure nucleus was identified on the fracture surface, the two measurements were made to the nearest 0.01 in. In subsequent analysis of the data, longitudinal measurements were referred to the axis representing the line of maximum stress. The transverse measurements were converted to the distance from the nearest corner of the specimen. Each such corner distance was divided by half the width of the specimen at the pertinent longitudinal section. These ratios expressed in per cent represented the corner distance or transverse location of the fatigue nucleus.

Following these measurements, the fracture surface of each specimen was

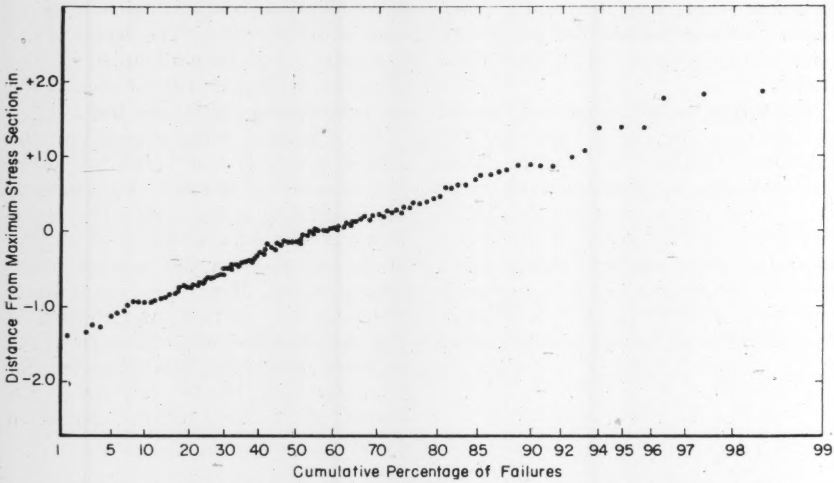

FIG. 3.—Cumulative Percentage of Failures to Indicated Longitudinal Distance for All Data.

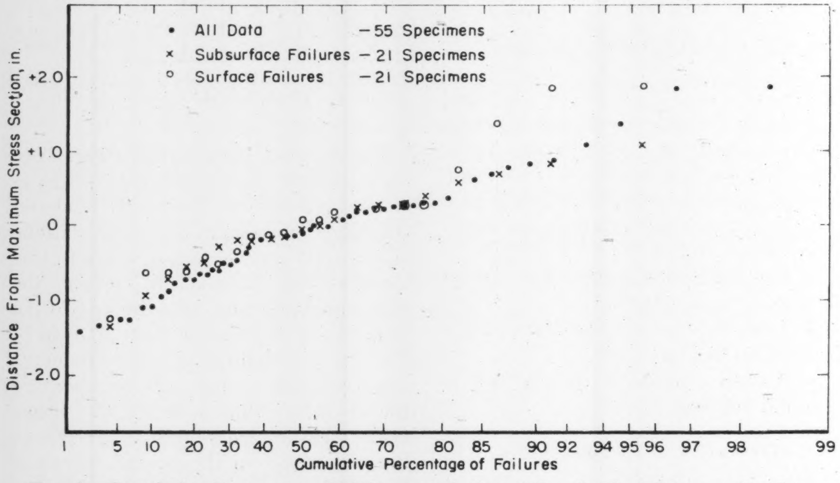

FIG. 4.—Cumulative Percentage of Failures to Indicated Longitudinal Distance for Groups Longitudinally Ground and Tested in Reversed Bending.

examined under low and moderately high magnification. When failure nuclei were observed below the surface of the specimen, the distance to the surface was measured to the nearest 0.0001 in. This inspection also disclosed that there was in many cases an inclusion at the nucleus. The dimensions of the inclusions were measured at high magnification both parallel and perpendicular to the surface of the specimen, and the size was expressed as the square root of the product of the two measurements.

LONGITUDINAL FAILURE DISTRIBUTIONS

Figures 3 and 4 are plots on extreme value probability paper of the cumulative percentage of failure to any position along the longitudinal axis of the specimen. The negative dimensions in these figures are in the direction of the *Y* axis of Fig. 1, measured from the maximum stress section. Figure 3 contains data from the 15 groups of bars investigated. However, data are included for only those specimens that were tested at stress levels in the range 90 to 114 per cent of their respective fatigue limits. Figure 4 contains data from all the groups of specimens longitudinally ground and tested in reversed bending. This grouping had the same number of surface and subsurface failures; hence, was useful in examining whether failure location (depth of nucleation) influenced the longitudinal fracture location.

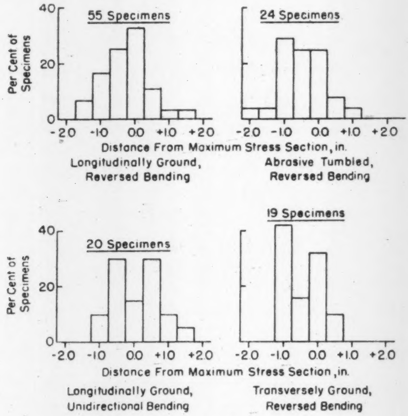

FIG. 5.—Histograms for Longitudinal Failure Locations for Four Groups of Processing and Testing Variables.

Figure 3 shows that all of the longitudinal failure data can be represented by a straight line over an appreciable range. Failures occurred over approximately 4 in. of the test section with the median location near the maximum stress section. About 70 per cent of the failures were in the region of the bar where the stresses were greater than 95 per cent of the maximum nominal applied stress. The remainder of the failures provide evidence that local inhomogeneities exist which raise the stress at the failure location 5 per cent or more to a value equal to or greater than the maximum nominal applied stress. The observation of inclusions at the fatigue nuclei, discussed later, supports the idea that metallurgical stress raisers are important.

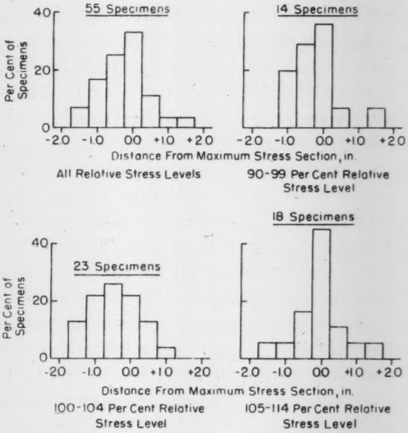

FIG. 6.—Histograms for Longitudinal Failure Locations for Longitudinally Ground Bars Tested in Reversed Bending.

The straight-line relationship is also evident in Fig. 4, but to a more limited extent. Also from Fig. 4, the longitudinal distribution of failure is not affected by the depth below the surface at which failure initiated.

In order to study the effect of some other variables on the longitudinal distribution of failures, histograms were constructed and are shown in Fig. 5 for each of the four major groupings of processing and testing variables. In the case of the longitudinally ground bars tested in reversed bending, histograms also were constructed for three ranges of the ratio of test stress to fatigue limit (denoted by relative stress level in Fig. 6 and in Table II). The three ranges indicated under the appropriate histogram in Fig. 6 were so selected that there would be approximately the same number of failed specimens in each group. It happened that the selected ranges also ordered, the data from the standpoint of survivors and failures. For example, in the stress range 105 to 114 per cent, all specimens tested were failures. In the range 100 to 104 per cent, about three fourths of the specimens were failures. Finally, in the stress range 90 to 99 per cent, about one third of the specimens tested were failures.

Since the number of specimens in some of the groups was relatively small, the histograms in Figs. 5 and 6 can be used only qualitatively. An examination of the figures shows that the distribution of longitudinal fractures was not affected by processing and testing variables, nor is stress level an influencing factor.

The failure distributions were analyzed further by plotting graphs of cumulative percentage of failure for each of these groups and by constructing confidence bands for each of the cumulative distributions. The method employed was that suggested by Kolmogoroff (3) for use when the cumulative distribution function is unknown as it is in these cases. The method also takes into account the differences in population of the various groups.

If this approach is used graphically in comparing two distribution functions and if the confidence bands plotted on the same graph overlap considerably, there would be no evidence for asserting that a difference exists in the two functions for the appropriate levels of confidence. If the confidence bands do not overlap, the evidence would indicate that there would be a real difference in the functions. Finally, if the confidence bands barely overlap, it would be necessary to use more powerful statistical tools to determine whether a difference exists. This approach confirmed the other more approximate analyses. Thus, it was established at the 95 per cent confidence level that there was no statistical evidence that the longitudinal failure location was influenced by (*a*) major processing and testing variables, (*b*) stress level, or (*c*) depth from the surface at which failure initiated.

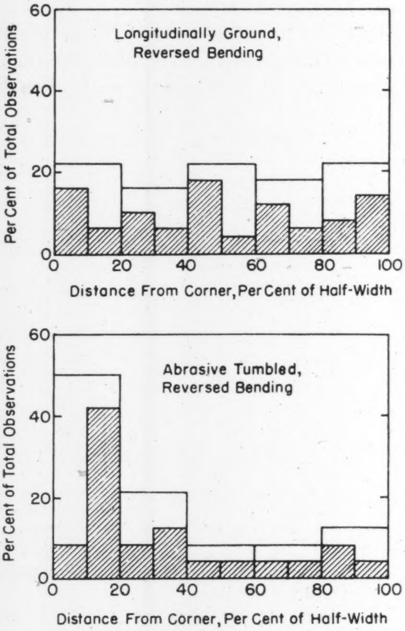

FIG. 7.—Histograms for Transverse Failure Locations for Two Groups of Processing and Testing Variables.

With reference to item (*b*), the conclusion is in agreement with statements of Cummings, Stulen, and Schulte (4, 5) who found qualitatively that there is an approximate stress level below which the scatter in failure location was high and single fatigue nuclei were observed on the fracture surfaces. The data considered in this paper probably were taken at stress levels appreciably below this approximate bounding stress level.

The effect of inclusion size on longitudinal location of failure also was examined. The data were displayed on a graph with coordinates of inclusion size and longitudinal distance. The graph is not shown since there was no evidence to suggest that the inclusion size or even the type of inclusion influenced the longitudinal failure location.

TRANSVERSE FAILURE DISTRIBUTION

It has been stated frequently (for example, reference (6)) that sharp corners can be detrimental and that fatigue cracks generally initiate at the corners of specimens having a rectangular cross-section. However, the fatigue failures in this study did not behave in this manner.

In Fig. 7 are shown histograms for two of the general processing and testing conditions. These include the groups of longitudinally ground bars and also the groups of abrasive tumbled bars, both tested in reversed bending. The former group was a homogeneous one that contained 55 specimens. The latter group had less than half that number of specimens. The histograms have been plotted for two increments of distance from the corner. The smaller increment, 10 per cent, represented by the cross-hatched areas, probably is too detailed for the number of specimens involved. In the histogram based on the larger 20 per cent increment, the ordinate is the sum of the ordinates of the two smaller increments within each larger unit of corner distance.

The histogram for the longitudinally ground specimens shows that the failure nuclei are distributed uniformly over the flat surface of the specimen, with no evidence that the corners were more frequent sites of fatigue nucleation.

A somewhat different conclusion might be deduced from the histogram for abrasive tumbled bars. In this case, the data show that the area adjacent to the corners provided a more frequent site for fatigue nucleation, even though the corners were rounded over in the tumbled bars and quite sharp in the ground ones. This indicates that the greater tendency of the abrasive tumbled bars to have failures initiate near the corners than away from it cannot be ascribed to the weakening effect of a corner.

These observations suggest that the specimen corners had little or no effect upon the location of failures. The fact that many specimens failed at inclusions remote from the corners indicates that the stress-raising influence of the inclusions was much more influential in determining the location at which failure nucleated.

Even though the corners were not particularly important as a discontinuity, it should be recognized that the fatigue limit is affected markedly by the shape of the cross section. This has been shown by several investigators (see, for example, references (7 and 8)).

DEPTH TO FATIGUE NUCLEUS

The fatigue nuclei were found either at the surface or somewhat below the surface (1, 2), with a large number having inclusions at the origin. If such an inclusion intersected the surface, the failure was called a surface failure. If the inclusion did not intersect the surface, the depth to the failure origin was considered as the distance from the surface to the center of the inclusion. For specimens that did not contain obvious inclusions at the nucleus, surface or subsurface nucleation was estimated from the appearance of the fracture surface.

For each group (corresponding to each processing condition) the fatigue nuclei seemed to be predominantly either at the surface or appreciably below the surface. For example, subsurface failures were generally observed in specimens ground with a combination of a moderately soft wheel and grinding oil. Similar failures were observed on the fracture surface of bars that were abrasive tumbled. Surface failures, however, were associated usually with grinding conditions that involved the use of either soluble oil or hard wheels. The bars ground transversely with soluble oil were an exception since they all failed below the surface.

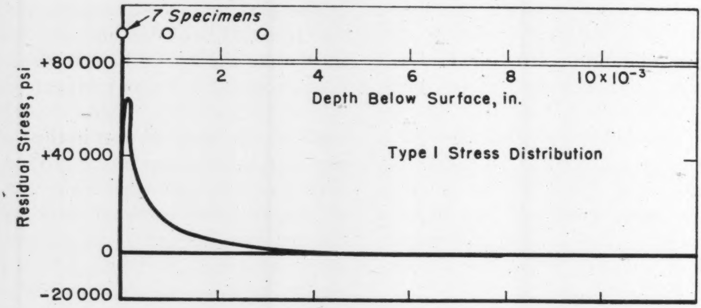

FIG. 8.—Residual Stress Distribution and Location of Fatigue Nuclei in Group BC Bars, Ground with Soft Wheel and Soluble Oil.

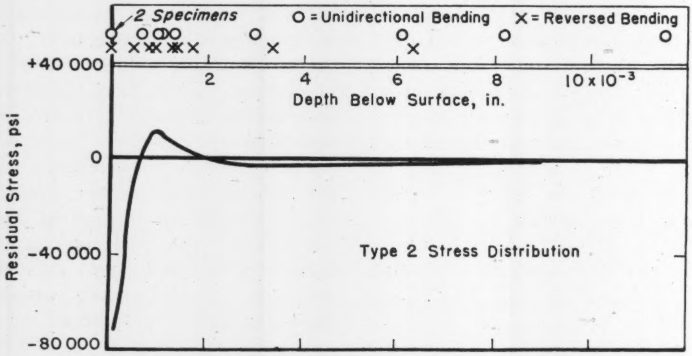

FIG. 9.—Residual Stress Distribution and Location of Fatigue Nuclei in Group AG Bars, Ground with Soft Wheel and Grinding Oil.

The experimental evidence indicated that the fatigue limits were higher for groups of bars for which failure initiated below the surface. While it is certain that some of the improvement in behavior could be attributed to compressive residual stresses, it is believed that surface cold work from grinding and tumbling also are important.

If the residual stress data and the failure depth data were considered together, several features of interest emerge. Figures 8, 9, and 10 show graphs of the three types of residual stress distributions, previously identified (1), on which are superimposed positions of the failure nuclei for each appropriate group.

From Fig. 8, when the residual stress is tensile, both at the surface and below, most of the failures initiated at the surface. Occasionally, a failure initiated below the surface at some underlying inclusion. Figure 9 shows that for those groups with moderate surface compressive stresses and subsurface tensile stresses and with fatigue limits that were appreciably higher than the base-line value, a large number of failures were observed at a range of depths where the tensile stresses are present and where the stress gradient is steep. A few surface failures were observed, and, once again, failures also initiated at inclusions located at appreciable depths below the surface.

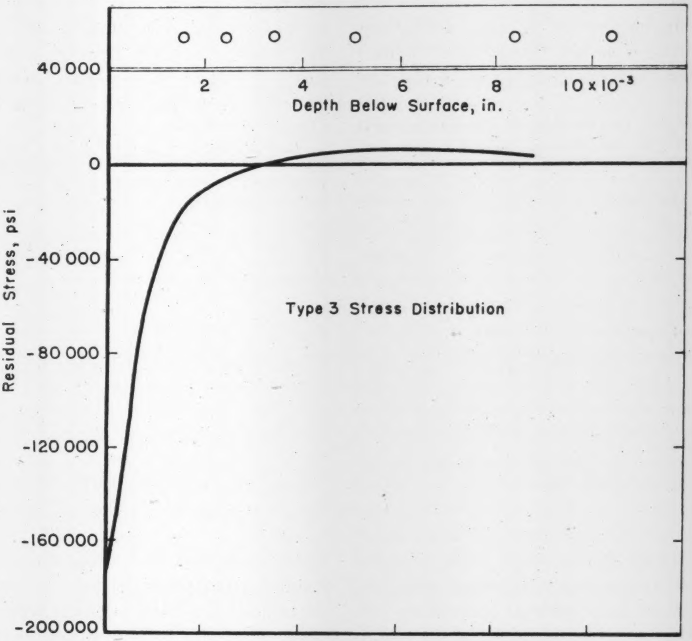

FIG. 10.—Residual Stress Distribution and Location of Fatigue Nuclei in Group AH Bars, Loose Tumbled with No. 0 Fused Aluminum Oxide Chips.

For specimen groups with very high surface compressive stresses and very low subsurface tensile stresses, as in Fig. 10, subsurface nucleation was found at depths where the residual stresses are negligible. However, once again, nuclei were observed at depths much below the residually stressed layer.

The inclusions at the fatigue nuclei had finite dimensions, and hence it may be more correct to say that failure initiated in an area where the residual stresses were changing rapidly. For some cases, this area included the surface as for groups shown in Figs. 8 and 9. In other cases, particularly tumbled bars, the surface was not included in the area where failure initiated.

The incidence of failures at significantly large depths below the surface indicates that residual stress is not the only factor of importance. Inclusions at such fatigue nuclei must have resulted in more unfavorable stresses than the residual stresses present near the surface. The variation in depth of inclusions at which failure initiates in a group of identically processed bars probably accounts for some of the scatter in the fatigue results.

TABLE I.—COMPARISON OF SIZE AND DEPTH OF INCLUSION TYPES.

Quantity	Oxide Inclusion	Titanium Cyanonitride Inclusion
Average Size,[a] in......	0.0037	0.0006
Range in Size, in......	0.0004 to 0.0193	0.0002 to 0.0035
Average Depth,[b] in.....	0.0041	0.0011
Range in Depth, in.....	0.0001 to 0.0146	0.0004 to 0.0035

[a] Size is expressed as the square root of the product of the transverse width and depth of each inclusion.

[b] Depth is expressed as the distance from the surface to center of inclusion.

INCLUSIONS

A limited examination of inclusion content was made using method A of ASTM Recommended Practice E 45.[4] The results indicated the inclusions to be predominantly the hard refractory-type oxides which were in most cases the alumina type, probably iron aluminate. The inclusion rating of the alumina type oxides was determined to be B-2 thin while the inclusion rating of globular type oxides (primarily iron oxides) was determined to be D-2 thin. These ratings represent inclusions approximately 0.0003 in. in diameter. However, as indicated subsequently, oxide inclusions and titanium cyanonitride inclusions of larger size were observed frequently at fatigue nuclei. The limited study apparently was not detailed enough to disclose the less numerous but larger size inclusions which were important from the fatigue standpoint; however, it gave an indication of the background inclusion level.

TABLE II.—PER CENT OCCURRENCE OF INCLUSIONS AS A FUNCTION OF RELATIVE STRESS LEVEL AND LIFETIME RANGE.

Relative Stress Level, per cent	Lifetime Range, cycles	Per Cent Occurrence	
		Oxide Inclusions	Titanium Cyanonitride Inclusions
90 to 99	...	19	14
100 to 104	...	54	58
105 to 114	...	22	24
> 114	...	5	4
...	10^4 to 10^5	8	4
...	10^5 to 10^6	27	17
...	10^6 to 10^7	65	79

In assessing the effect of inclusions on fatigue, only the subsurface failures were studied in detail. Of the 75 failures examined, 70 failures had inclusions at the origin. About 55 per cent of the inclusions were of the complex oxide type, the remainder being titanium cyanonitride inclusions. The subsurface data were examined further with the objective of determining whether the two kinds of inclusions had the same effect on fatigue behavior.

The inclusions at which failures occured differed markedly with respect to size and depth below the surface, as shown in Table I. On the average, the oxide inclusions were about six times as large as the others and were located about four times as far from the surface. In spite of these differences, the relative occurrence of the two types of inclusions at the fatigue nuclei was not affected by the stress level or by the fatigue lifetime, as is evident from Table II.

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[4] Recommended Practice for Determining the Inclusion Content of Steel (E 45–51), 1955 Book of ASTM Standards, Part 1, p. 1645.

When each type of inclusion was considered separately, a similar analysis showed that the relative stress level or fatigue lifetime did not have any detectable influence on the size and depth of the inclusions associated with the fatigue nuclei.

CONCLUDING REMARKS

The study of the distribution of failures in hardened steel fatigue bars has indicated a number of interesting features:

1. The longitudinal distribution of failures was insensitive to the major processing and testing variables studied in this program and to the relative stress levels examined and to the depth at which failure initiated. The processing variables included longitudinal grinding, transverse grinding, and abrasive tumbling. The testing variables were reversed and unidirectional bending.

2. The longitudinal failure distribution, when plotted on extreme value probability paper, could be approximated by a straight line, with a median value near the maximum stress section.

3. The transverse failure distribution did not appear to be affected by processing or testing variables. The data indicated that there was little or no tendency for the fatigue failures to originate at the corners of the specimens, contrary to what might be expected from some of the literature. The evidence suggested that inclusions remote from the corners were as detrimental.

4. The depth of nucleation appears to be related to processing variables. Since many of the failures initiated at inclusions which were of a finite size, most failures could have initiated in areas where the residual stresses were changing rapidly. As this transition layer became deeper, resulting from specific processing conditions, failures generally were at greater depths.

5. Two kinds of inclusions generally were found at the fatigue nuclei: complex oxide inclusions and titanium cyanonitride inclusions. The oxide inclusions were more massive than the latter and were found at considerably greater depths from the specimen surface. There was no evidence, however, that the larger oxide inclusions affected fatigue behavior differently than did the titanium cyanonitride inclusions.

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(3) A. Kolmogoroff, "Confidence Limits for an Unknown Distribution Function," Annals of Mathematical Statistics, Vol. 12, pp. 461–463 (1941).

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(5) H. N. Cummings, F. B. Stulen, and W. C. Schulte, "Investigation of Materials-Fatigue Problems Applicable to Propeller Design,"

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(7) L. P. Tarasov and H. J. Grover, "Effects of Grinding and Other Finishing Processes on the Fatigue of Hardened Steel," *Proceedings,* Am. Soc. Testing Mats., Vol. 50, pp. 668–698 (1950).

(8) T. J. Dolan, J. H. McClow, and W. J. Craig, "The Influence of Shape of Cross Section on the Flexural Fatigue Strength of Steel," *Transactions,* Am. Soc. Mechanical Engrs., Vol. 72, No. 5, pp. 469–476, July, 1950.

DISCUSSION

MR. H. E. FRANKEL.[1]—Although the authors have emphasized the importance of processing variables on the depth of nucleation of fatigue fractures, I would like to point out that composition may perhaps be of equal importance.

Data obtained at the National Bureau of Standards on experimental heats of steels containing 1.3 per cent copper showed that two thirds to three fourths of the specimens tested gave a subsurface nucleation effect. As these specimens had been processed in a manner similar to thousands of other specimens, the inference is that the copper is the agent responsible for the large number of failures initiating at inclusions below the surface.

MR. W. S. HYLER (*author*).—Mr. Frankel has raised an interesting point in suggesting that composition may be important in affecting the depth of nucleation of fatigue fractures. It is possible in this case that the addition of 1.3 per cent copper could have influenced the cold-working characteristics of this steel to such an extent that the characteristics of the surface layer were sufficiently altered to promote subsurface nucleation. Thus, although similar surface preparation techniques may have been used, more favorable residual stress distributions may have been formed in the surface layer and the layer may have been cold worked more extensively in the steel with 1.3 per cent copper. Each of these effects could lead to a strengthened surface layer which could lead to subsurface nucleations at inclusions.

MR. T. J. DOLAN.[2]—I think the authors should be congratulated on doing a very tedious and painstaking job of trying to locate the nuclei for failure in specimens of this nature. Those who have looked at a variety of fractured surfaces can realize the complexity of the task involved.

In several tests over recent years we have had occasion to watch the development of cracks in the surfaces of rather large specimens in which a surface zone having a length of 4 to 6 in. was all within 95 per cent of the peak stress. In these instances we were amazed to find there was no such thing as a *single* fatigue crack; in general there were hundreds of fatigue cracks. The cracks that one sees are often the final development of groups of microscopic voids which grow and join together; at a late stage these become of visible size and frequently many of these (which grow at different rates) become visible to the eye before fracture. In one instance some specimens about $1\frac{1}{2}$ in. wide were tested with the zone of high stress over a length of about $\frac{1}{2}$ in. on the specimen. In these cases when the specimen had been cycled for about two thirds of its fatigue life, one could count as many as 25 to 30 separate and distinct visible cracks in the surface if careful observational procedures were used.

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[1] National Bureau of Standards, Washington, D. C.

[2] Head, Department of Theoretical and Applied Mechanics, University of Illinois, Urbana, Ill.

This brings me to the question—just what does one use as a criterion of where the fatigue crack starts when the early stages of failure consist of dozens of cracks in small zones or hundreds of cracks in large zones? This question also applies to the paper presented by Mr. Stulen.[3] How does one decide exactly where "the crack" starts when oftentimes a fatigue fracture may consist of many cracks, starting in irregularly spaced zones, growing at irregular rates with the accumulation of ruptured surfaces becoming a rather complex statistical problem?

MR. HYLER.—In our work we have not attempted specifically to count numbers of cracks prior to failure. We have examined the specimens after failure and in this examination have generally found no other cracks on the surface and no more than one fatigue nucleus on the fracture surface. From this we assume that the specimens had only one crack.

As far as determining the specific starting point of a fatigue crack on a given plane, this is a more difficult question, particularly for those cases where failure is associated with an inclusion that has some rather finite dimensions. In most of our work the region of the residual stress diagram where the stress gradient was steepest is the region in the specimen where failure probably started. However, failure could have initiated at the top, the bottom, or at any spot along the sides of an inclusion in this region. However, as Mr. Dolan points out, failure could have started at more than one site in the vicinity of the inclusion and as the cycle life increased these small fissures may have coalesced and grown to final fracture. Perhaps Mr. Stulen has some remarks on this, too.

MR. F. B. STULEN.[4]—In our work we found both situations can exist. It really depends on the stress level at which you are testing. At low stress levels very close to the endurance limit there seem to be just a few cracks that start and propagate sufficiently rapidly to dictate the fracture surface. On the other hand, at high stresses you find a multiplicity of cracks that are forming and joining and eventually causing the fracture surface. So I think it really depends on the stress level that you are dealing with as to whether you get a single type nucleus or whether you get multiple nuclei in the fractured surface.

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[3] H. N. Cummings, F. B. Stulen, and W. C. Schulte, "Tentative Fatigue Strength Reduction Factors for Silicate-Type Inclusions in High-Strength Steels," p. 505, this publication.

[4] Assistant Chief Engineer, Curtiss-Wright Corp., Caldwell, N. J.

EFFECT OF STATIC PRESTRAIN ON THE FATIGUE PROPERTIES UNDER UNIFORMLY INCREASING STRESS AMPLITUDE*

By F. H. Vitovec[1]

Synopsis

A study was conducted on the effect of short-time and creep prestrain on the fatigue properties of unnotched and notched specimens of alloys 7075-T6 and 2024-T4 extruded, 16-25-6 hot-cold-worked, and S-816 solution treated and aged. Fatigue properties were determined using a progressive load fatigue test with a single loading rate of 0.01 psi per cycle. The unnotched and notched specimens were prestrained at test temperature and then immediately subjected to a fatigue test at the same temperature and in the same testing machine. The test temperatures for the short-time prestrain and creep prestrain respectively were 75 and 300 F for 7075-T6, 300 and 500 F for 2024-T4, 1200 F for 16-25-6, and 1500 F for S-816. Short-time prestrains up to 10 per cent were applied. Stresses used for the creep prestrains corresponded to those which produced rupture in 100 to 200 hr at test temperature.

The main factors influencing the fatigue failure stress were residual stresses, metallurgical reactions, and cracks produced by tensile prestraining. Strain hardening and relaxation of residual stress appeared to be of secondary importance for the materials and the range of testing variables investigated.

Uncontrolled permanent strain of a structural part due to unidirectional loads may occur during manufacture, assembly, and service. Such permanent strain, which may occur either locally or extend over the entire part, can have either detrimental or beneficial effects on the mechanical behavior under service conditions.

Earlier studies of the effect of prestrain on fatigue properties were primarily concerned with the effect of coldwork and residual stress on the fatigue properties at room temperature. In this regard the effect of shot peening and surface rolling on the fatigue properties was most intensively studied. At elevated temperatures, the effect of static prestrain and the fatigue loads on metallurgical changes become equally important. The duration of static load application is particularly important at elevated temperatures. Thus, short-time prestrain may have a different effect on the fatigue properties than long time, or creep prestrain.

This study is concerned with the effect of short-time and creep prestrain on the fatigue properties under uniformly increasing stress amplitude.

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* Presented at the Sixty-first Annual Meeting of the Society, June 22–27, 1958.

[1] Department of Mechanics and Materials, University of Minnesota, Minneapolis, Minn.

TABLE I.—TESTING PROGRAM.

Test Material	Unstressed Time at Temperature, deg Fahr		Short-Time Prestrain					Creep Prestrain					
			Unnotched			Notched		Unnotched			Notched		
	Unnotched	Notched	Temperature, deg Fahr	Starting Stress	Type of Prestress	Temperature, deg Fahr	Starting Stress	Temperature, deg Fahr	Creep Stress	Starting Stress	Temperature, deg Fahr	Creep Stress	Starting Stress
7075-T6 aluminum alloy	...	...	75	22 000	Tension-compression	75 75	7000; 5000 7000; 5000	300	52 000	15 200	300	52 000	7 000
2024-T4 aluminum alloy	500	500	300	22 000	Tension-compression	300 300	12 000 7000; 5000	300 500	64 000 20 000	22 000 15 200	500	20 000	7 000
S-816 alloy	1500	...	1500	26 000	...	...	...	1500	26 000	26 000	...	...	...
16-25-6 Timken alloy	1200	1200	1200	41 400	Tension	1200	15 020	1200	4 500	41 400	1200	55 000	15 200

TABLE II.—CHEMICAL COMPOSITION AND MECHANICAL PROPERTIES OF TEST MATERIAL.

Test Material	7075-T6	2024-T4	16-25-6	S-816
Chemical composition, per cent	1.57 copper, 0.20 iron, 0.12 silicon, 0.02 manganese, 0.23 chromium, 2.46 magnesium, 5.63 zinc, 0.02 titanium.	4.30 copper, 0.22 iron, 0.14 silicon, 0.61 manganese, 1.54 magnesium, 0.01 zinc, 0.01 chromium, 0.01 titanium.	0.102 carbon, 1.92 manganese, 0.528 silicon, 0.009 sulfur, 0.01 phosphorus, 16.45 chromium, 27.0 nickel, 6.60 molybdenum, 0.133 nitrogen, Iron balance.	0.397 carbon, 1.12 manganese, 0.50 silicon, 0.018 sulfur, 0.012 phosphorus, 19.42 chromium 20.62 nickel, 4.1 molybdenum, 42.9 cobalt, 2.99 iron, 2.86 columbium, 1.03 tantalum, 4.03 tungsten.
MECHANICAL PROPERTIES OF TEST MATERIAL AT ROOM TEMPERATURE				
0.2 per cent yield strength, psi	84 000	70 500	80 000	74 000
Tensile strength, psi	92 000	88 000	120 000	147 000
Elongation, per cent	10	11	13	23
Hardness	Rockwell 87–89 B	Rockwell 81 B	269–311 BHN	Rockwell 27–28 C
Fatigue strength,[a] psi:				
$K_t = 1.0$	29 000	26 000	54 000	56 000
$K_t = 3.4$	11 000	10 000	19 000	23 500
MECHANICAL PROPERTIES OF TEST MATERIAL AT TEST TEMPERATURE[b]				
Test temperature, deg Fahr	75	300	1 200	1 500
0.2 per cent yield strength, psi	...	60 000	77 000	50 000
Tensile strength, psi	...	75 000	93 000	76 000
Elongation, per cent	...	15	13	12
Fatigue strength, psi:				
$K_t = 1.0$	...	...	42 200	37 600
$K_t = 3.4$	...	...	19 200	18 400

[a] Reversed direct stress fatigue strength at 2.16 × 10^7 cycles. (Conventional constant stress amplitude tests.)

[b] Specimens used are different from standard test specimens.

TEST PROGRAM AND MATERIALS

The testing program is shown in Table I. The unnotched and the notched specimens were prestrained at the indicated temperature and then immediately subjected to a progressive load fatigue test (**1, 2, 3**)[2] at the same testing temperature and in the same testing machine. Fatigue tests with uniformly increasing alternating stress amplitude were used instead of conventional constant stress amplitude tests in order to reduce the number of tests. This facilitated the study of the effect of prestrain over a wide range of strain.

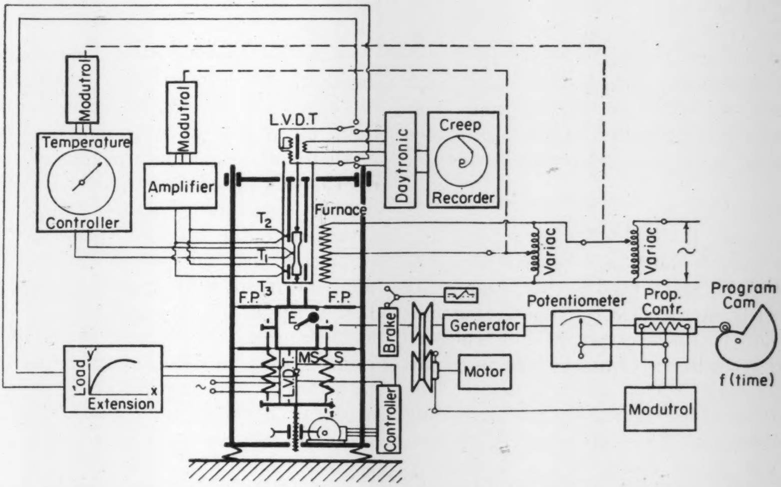

FIG. 1.—Schematic Diagram of Direct Stress Fatigue and Creep Testing Machine with Load Programming Device, Furnace with Temperature Controlling System, and Load-Extension, and Strain-Time Recording Devices.

The fatigue tests were performed with a single and relatively low rate of stress amplitude increase. The failure stress so determined is used to indicate the trend of the effect of prestrain on the fatigue strength.[3]

Also indicated in Table I is the stress for creep prestrain and the starting stress for the fatigue tests for each material.

Two light alloys and two heat-resistant alloys were used for this investigation. The aluminum alloys 2024-T4 and 7075-T6 were in the form of extruded bars. The materials were from the same batch as those used in a previous study of fatigue properties under combined alternating and static stress (**4**). The details on fabrication, metallographic structure, etc., are given in reference (**4**).

The heat-resistant alloys 16-25-6, Timken and S-816 were from the same heat as those used in a study of fatigue, creep, and rupture properties (**5**). The Timken alloy 16-25-6 was "hot-cold worked" according to Fleischmann (**9**).

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[2] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 568.

[3] This non-standard fatigue testing method was previously used by the author to indicate the stress range in an extensive standard fatigue testing program including 9 different alloys at room temperature and elevated temperature. In these tests the failure stress varied consistently with the fatigue strength at the corresponding number of cycles to fracture.

The chemical composition and the mechanical properties of all four alloys are listed in Table II.

Two types of cylindrical axial stress specimens were used. The unnotched specimen (stress concentration, $K_t = 1$) had a test section 0.5 in. long and 0.2 in. in diameter. The notched specimen ($K_t = 3.4$) had a minimum diameter of 0.25 in. The 60-deg notch was machined to a depth of 0.062 in. with a notch radius of 0.01 in.

In view of the significance of cold working and residual stresses produced by machining considerable care was taken in specimen preparation. The specimen preparation procedure is described in detail in references (5) and (6).

TESTING EQUIPMENT AND TEST PROCEDURE

Figure 1 shows a schematic diagram of the test setup used in this program. It consists of the direct stress testing machine, the furnace control and temperature recording system, the load programming and speed control device, the load-extension indicating and recording system, and the creep recording system.

The direct stress fatigue and creep testing machine is similar to that described in reference (7). The alternating force is produced by the eccentric, *E*. The eccentric is driven by a variable speed transmission which permits a change in speed and hence of the alternating force. The speed of the eccentric is electronically controlled; it is determined by the program cam which, for the tests of the present program, is designed to produce a loading rate of 0.01 psi per cycle. Attached to the driving shaft of the eccentric is a brake actuated at fracture of the specimen in order to stop the driving mechanism quickly and reduce damage of the fracture surface.

The static load is applied by two springs, *S*. For creep tests the static load is maintained constant by holding a constant spring deflection with a control device.

The specimens are heated in a long electric resistance furnace with windings spaced further apart in the test section and closer at the ends, thus maintaining uniform temperature over the test section of the specimen. The main temperature control is operated by the thermocouple T_1 which senses the temperature in the center of the test section of the specimen.

Since the heating conditions change during the test due to creep extension of the specimen, material damping, vibrations, etc., a second controlling device is used to maintain a uniform temperature distribution along the specimen. This is done by two thermocouples, T_2 and T_3, attached to the specimen grips and so connected that no current flows if the temperature of both grips is the same. If the grips are at different temperatures a current results and this, after amplification, is used to drive another variable transformer which adjusts the voltage across each half of the furnace winding.

The extensometer is attached to the specimen ends and indicates the extension of the specimen by means of a differential transformer (5). The output of the differential transformer is either fed into a creep recorder or into an *x-y* plotter for the recording of load-extension diagrams of short-time tension tests. The static load is indicated by the spring extension by means of a second Schaevitz differential transformer which is connected to the *y*-scale of the *x-y* plotter.

The core of the differential transformer is attached to the extensometer by a calibrated screw which permits rapid calibration of the extensometer at test temperature before each test.

This equipment permits fatigue tests to be conducted immediately following either short-time static, creep tension, or

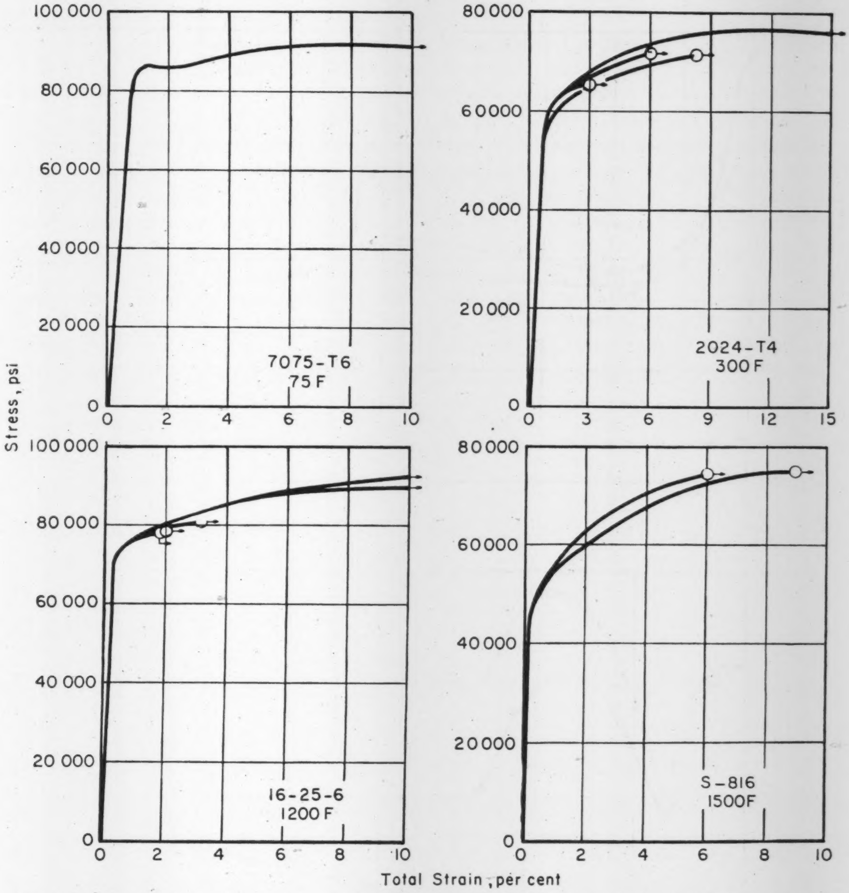


FIG. 2.—Stress-Strain Curves Recorded During Prestrain of the Alloys 7075-T6, 2024-T4, 16-25-6, and S-816.

compressive prestrain at the test temperature. This appears to be very important for precipitation-hardening alloys for which intermediate cooling may affect the mechanical properties.

The heating period to equilibrium conditions was 45 min for the 2024-T4 aluminum alloy and 1 hr for 16-25-6 Timken and S-816 alloy.

Following the heating period, prestrain was applied which was immediately followed by the fatigue test at the same temperature. The time interval between the end of prestraining and the fatigue test was 15 to 20 min.

Load extension curves and creep curves were recorded during prestraining of the unnotched specimens.

Unnotched specimens were prestrained by short-time unidirectional load applica-

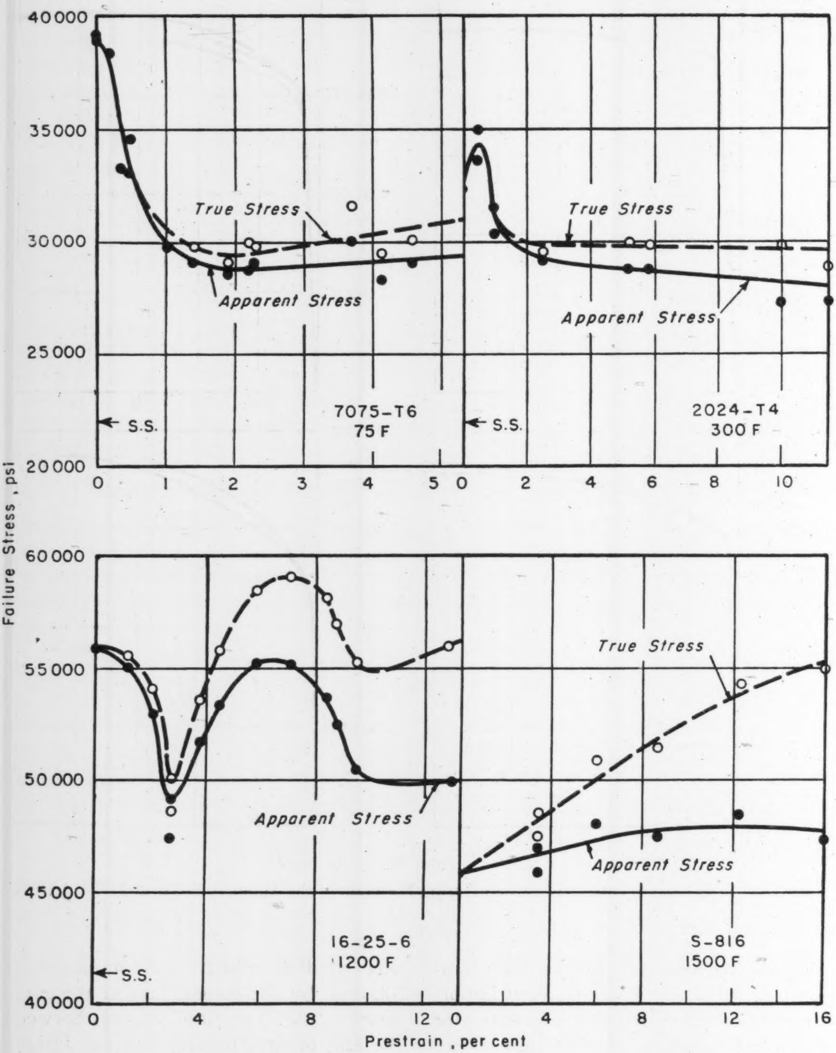


FIG. 3.—Effect of Short-Time Static Tensile Prestrain on the Fatigue Failure Stress of Unnotched Specimens.

S.S. = Starting Stress, Apparent Stress-Load divided by original cross-section. True Stress-Load divided by instantaneous cross-section.

tion within a range from a few tenths of a per cent to strains at ultimate stress. The time for "short-time" prestraining was from 5 to 10 min. Creep prestrain was performed at stresses which would have caused fracture within a period of 100 to 200 hr.

The fatigue test was started by increasing the motor speed from zero to 1800 rpm in approximately 5 sec. The uniform (symmetrical) increase of the tensile and compressive stress amplitude to that of the starting stress is very significant in analyzing the effect of fatigue stress on residual stresses produced by prestraining. From 1800 rpm on, the controlled speed was further increased so that an increase in stress amplitude of 0.01 psi per cycle resulted. The maximum motor speed at fracture was approximately 3100 rpm. Frequency effects due to variation of the motor speed within the indicated range can be considered negligible.

The number of cycles to fracture ranged from about 2.5×10^6 to 3.5×10^6 cycles.

EFFECT OF SHORT-TIME PRESTRAIN ON THE FATIGUE PROPERTIES OF UNNOTCHED SPECIMENS

Stress-strain diagrams as recorded during prestrain for the four alloys at test temperature are shown in Fig. 2. The effect of short-time static prestrain on the fatigue failure stress of the four alloys is shown in Fig. 3.

The data for 7075-T6 aluminum alloy indicate a significant decrease of fatigue failure stress by plastic prestrain at room temperature. A reduction of 25 per cent occurred at 2 per cent tensile prestrain. A similar reduction of failure stress resulted from compressive prestrain.

In order to study the cause of the decrease of fatigue strength, several small specimens of the 7075-T6 alloy with a cross-section of 0.10 by 0.03 in. were prestrained in a micro-tension testing machine, which is described in reference (6), and the deformation was observed during straining under the microscope. Stressing in the elastic range did not cause any observable initiation of cracks. However, with the onset of plastic deformation very small cracks were formed which grew in number and size with increasing deformation. Although the investigation did not furnish evidence that the cracks initiate at inclusions, it is very likely so.

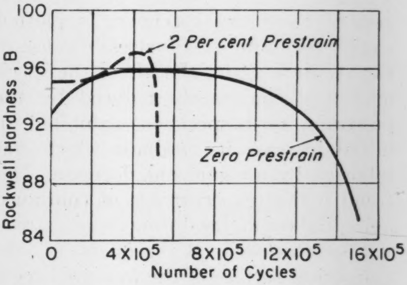


FIG. 4.—Rockwell Hardness B of 7075-T6 Aluminum Alloy as Function of Number of Cycles at 22,000 psi Reversed Direct Stress. (Hardness determined on flat specimen.)

The microscopic study was extended to regular size test specimens in the fatigue testing machine. The specimens were inspected for cracks after static prestrain and after various numbers of cycles of fatigue stress. Static prestrain caused a similar crack pattern as observed with the micro-test specimens. Fatigue stress caused a growth of those cracks that were perpendicular to the stress direction.

Although all evidence points towards initiation of cracks by perstrain as a cause of the severe reduction of fatigue strength, there may be other factors superimposed on this effect as emphasized by the fact that similar reductions were found in specimens with compressive prestrain. One of these may be localized continued aging and overaging during fatigue stressing, thus creating "soft spots" in which fatigue may proceed faster. Prestrain may accelerate this process and thereby reduce the failure stress.

The Rockwell B hardness of a 7075-T6 specimen with rectangular cross-section was measured periodically during fatigue cycling at 22,000 psi constant stress amplitude. Another specimen was 2 per cent tensile prestrained and then subjected to the same fatigue stress. Hardness of these two specimens is plotted *versus* the number of stress cycles in Fig. 4. Here again, the life of the specimen was significantly reduced by the prestrain. Both specimens exhibited an initial increase in hardness which was followed by a significant decrease. This trend indicates occurrence of continued aging and overaging during fatigue stress.

The Rockwell B hardness of the cylindrical surface of the fractured regular specimens was also measured. The hardness at 30 days after fracture is plotted *versus* prestrain in Fig. 5. Prestrain apparently accelerated the aging process resulting from fatigue stress, which results in a decrease of hardness with increasing prestrain.

These observations indicate that both effects, cracking during prestrain and overaging during fatigue stress, together cause the significant decrease of fatigue strength due to short-time prestraining.

The diagrams of Fig. 3 also show the effect of short-time tensile prestrain on the failure stress of the 2024-T4 aluminum alloy at 300 F. Prestrain causes an initial increase in failure stress, a maximum at about 0.5 per cent prestrain, and a decrease of failure stress of about 10 per cent for prestrains larger than 1 per cent. This trend may indicate the occurrence of precipitation processes influenced by prestrain. The initial increase may be explained by continued precipitation hardening. The decrease in failure stress resulting from larger prestrain may be attributed to overaging. No cracking during prestrain, as exhibited by 7075-T4 alloy, could be observed with this material.

Permanent tensile prestrain of the 16-25-6 Timken alloy at 1200 F decreases the fatigue failure stress at first. A maximum reduction of approximately 16 per cent occurs at about 2.7 per cent prestrain. Prestrains larger than 2.7 per cent cause an increase of the failure stress from the minimum to values as high as those exhibited by the unstrained material. This increase in failure stress indicates that the initial decrease is very probably caused by metallurgical factors and not by initiation of cracks during prestrain. The entire trend of the curve appears to be caused by reversion from age hardening and renewed precipitation, similar to that observed in aluminum-copper alloys (10).

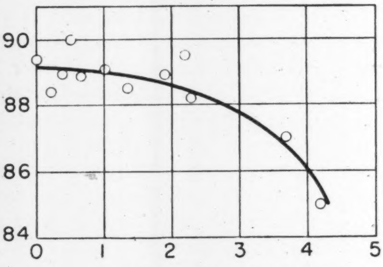


FIG. 5.—Effect of Prestrain on Rockwell Hardness B of 7075-T6 Aluminum Alloy After Prot Fatigue Test and 30 Days Aging at Room Temperature. (Hardness determined on fractured cylindrical specimen.)

Tensile prestrain of the S-816 alloy at 1500 F increases the fatigue failure stress significantly.

EFFECT OF PRIOR CREEP ON THE FATIGUE PROPERTIES OF UNNOTCHED SPECIMENS

A number of previous studies were concerned with the effect of prior creep on mechanical properties other than fatigue, such as notch impact strength (**11–14**), yield strength, ultimate strength and ductility (**14–16**), and retained rupture properties (**17**). In most cases a decrease in ductility and strength due to prior creep was observed.

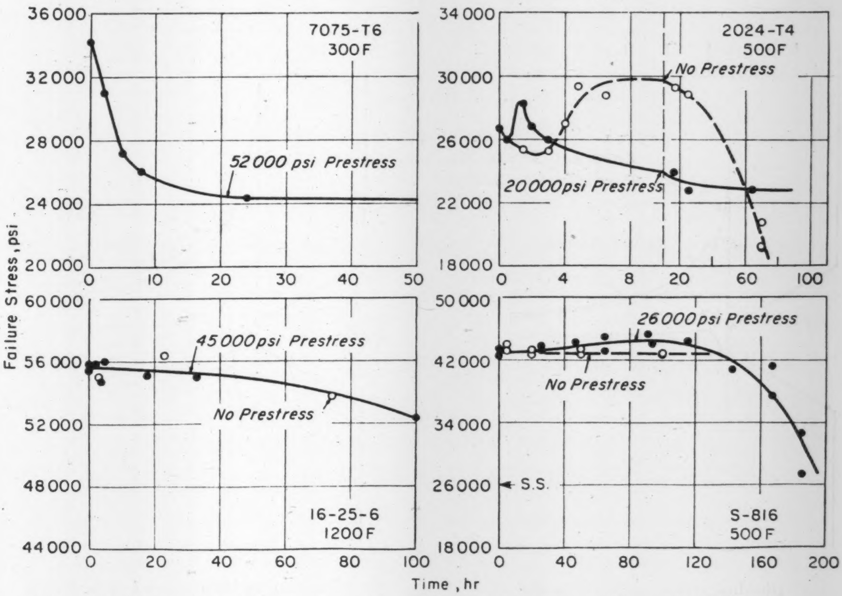


FIG. 6.—Effect of Creep Prestrain on the Fatigue Failure Stress of Unnotched Specimens. S.S. = Starting stress.

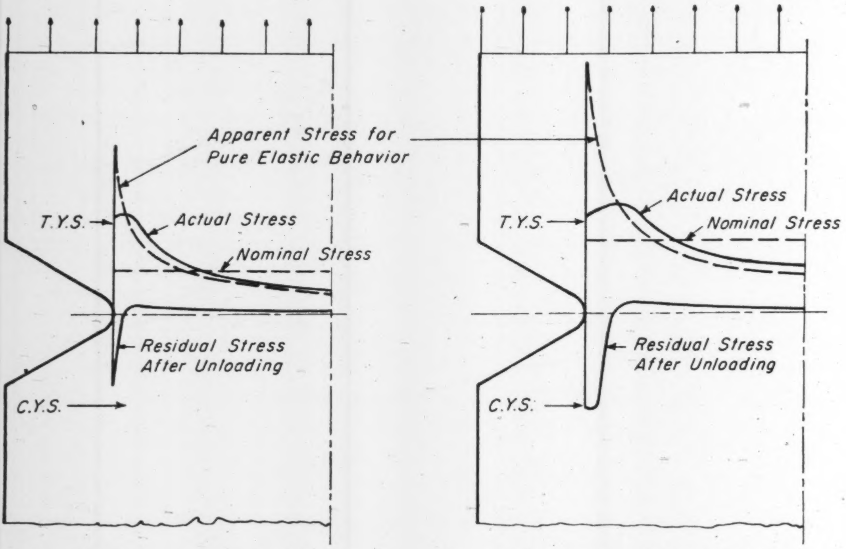
The test temperature of 300 F for creep prestrain of 7075-T6 was selected because the Rockwell B hardness of this material was not affected by extended exposure to this temperature, whereas a significant decrease in hardness occurs at 500 F.

Prior creep of 7075-T6 at 300 F and a stress of 52,000 psi causes a significant reduction of the failure stress, as shown by the diagram of Fig. 6. The trend is similar to that observed for short-time prestrain at room temperature and is probably caused by the same mechanism, that is, initiation of cracks during prestrain and aging effects.

The effect of prior creep on unnotched specimens of the 2024-T4 aluminum alloy at 300 and 500 F is characterized by aging reactions. This was indicated by the variation of the DPH-microhardness (25 gm load) and the Rockwell B hardness with unstressed exposure time which was identical with the variation of the fatigue failure stress. Figure 6, for data taken at 500 F shows that unstressed exposure time at temperature and also prior creep at 20,000 psi cause an initial decrease of the failure stress. After reaching a minimum in a relatively short time the failure stress raises to a maximum beyond which it decreases to values smaller than the previously observed minimum. The alloy was solution treated and aged at room temperature for 5 yr. Both, heating at higher temperature than the previous aging temperature and prior creep cause a reversal of the aging process which results in a decrease in strength properties (10). Renewed aging under the test conditions causes an increase of the strength properties followed by a second decrease in strength which is caused by continued aging and overaging.

Comparison of the effect of unstressed aging at 500 F and that of prior creep at 20,000 psi on the failure stress indicates that prior creep accelerates the aging reactions, for example, the minimum and the maximum occur at shorter times.

The 16-25-6 Timken alloy is apparently little affected by prior creep at 1200 F and a stress of 45,000 psi within the time range investigated (Fig. 6). Unstressed time at temperature as well as prior creep cause a slight decrease of the failure stress with increasing time. No difference between the effect of time at temperature and that of prior creep is apparent. This indicates that aging reactions are dominant. It is interesting to compare these results with those of short-time prestrain (Fig. 3). Short-time prestrain also caused an initial decrease of the failure stress; however, a minimum occurred after which the failure stress increased to original values. A similar behavior would probably be observed for prior creep at the stress of 45,000 psi if



a) Residual stress smaller than the yield strength.

(*b*) Maximum residual stress is determined by local yield strength.

FIG. 7.—Schematic Illustration of the Stress Distribution During and After Prestress.

T.Y.S. = Tensile yield strength. C.Y.S. = Compressive yield strength.

the time were extended beyond the range investigated.

Time at temperature up to 100 hr did not affect the failure stress of unnotched specimens of S-816 alloy at 1500 F. Prior creep at the same temperature and at 26,000 psi caused a slight increase of the failure stress, up to a maximum at about 100 hr which is followed by a relatively rapid decrease as shown in Fig. 6. This trend is obviously caused by aging reactions occurring at this temperature, resulting from creep.

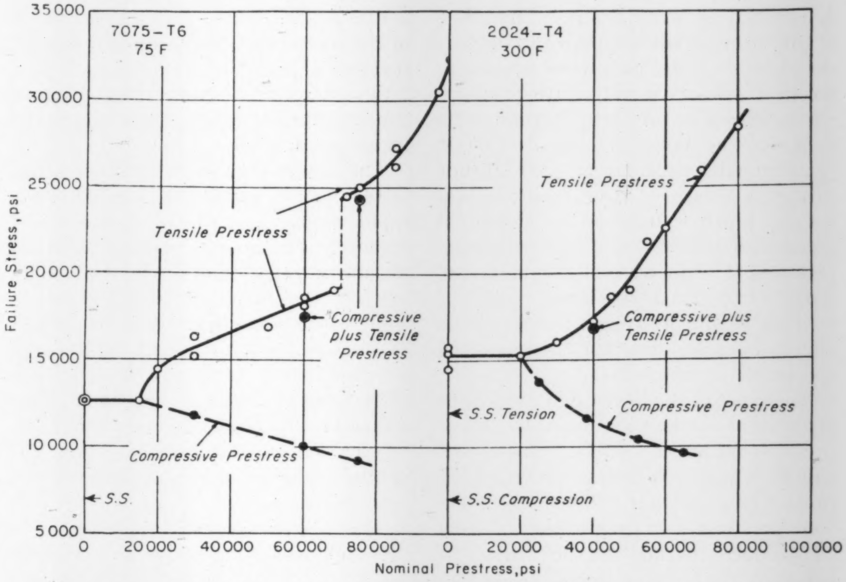


FIG. 8.—Effect of Short-Time Static Prestress on the Fatigue Failure Stress of Notched Specimens ($K_t = 3.4$).
S.S. = Starting stress.

EFFECT OF SHORT-TIME PRESTRAIN ON THE FATIGUE PROPERTIES OF NOTCHED SPECIMENS

The creation of residual stresses by prestressing of notched specimens is schematically illustrated in Fig. 7. It shows a case in which the maximum strain in the root of the notch exceeds the strain at yield stress. The resulting stress distribution caused by prestress is shown together with the theoretical stress distribution for pure elastic strain. Residual stresses remain after unloading from prestress as a consequence of the local plastic strain in the region of maximum strain. The residual stress distribution may be obtained approximately from the difference between elastic stress and actual stress. For case (*a*) it was assumed that the residual stresses are smaller than the yield strength. In this case the maximum stresses occur only in a very small volume and decrease rapidly with increasing distance from the surface. The depth over which the maximum residual stresses act increases if the residual strains exceed the strain at yield stress in the opposite strain direction, see case (*b*). The depth over which the residual stresses act may have a significant effect on the fatigue and relaxation behavior of the specimen.

In the diagram of Fig. 8 failure stress of notched specimens ($K_t = 3.4$) is plotted *versus* static nominal prestress (force divided by minimum cross-sectional area). The true prestress at the root of the notch can be approximately determined as nominal stress times K_t. If this stress is smaller than the proportional limit of the unnotched specimen, no effect is observed on the fatigue failure stress. Tensile prestress beyond this increases the failure stress. At 70,000 psi nominal tensile prestress an abrupt further increase of the failure stress occurs, which is followed by a gradual increase of failure stress with increasing prestress. This increase of fatigue failure stress of notched specimens with increasing tensile prestress is inverse to the effect of tensile prestrain on unnotched specimens (compare Fig. 3).

The cause of the increase of the fatigue strength of notched specimens by tensile prestraining appears to be primarily residual compressive stress produced by prestraining. Several notched specimens were prestressed in compression in order to produce residual tensile stress. As expected, the failure stress decreases with increasing nominal compressive prestress as shown in Fig. 8.

Two specimens were compressively prestressed and followed by a tensile prestress of the same magnitude. The failure stress of these specimens was close to that of specimens which were prestressed in tension only. This indicates not only that the weakening effect of compressive prestress can be removed to a large extent by subsequent tensile prestress, but also shows that the strain hardening effects are small as compared to those of residual stresses.

Notched specimens that were tension prestressed to about 100,000 psi nominal stress developed cracks along the circumference of the root of the notch during the application of the static load. These cracks apparently did not affect the failure stress. In some cases a crack was produced along the entire circumference of the notch by static tensile prestress. Due to the residual compressive stress acting on the crack, and the sharpness of the root of the crack, which restricted reversed slip and fatigue in this region, fatigue fracture occured along a coarse conical surface rather than along the transverse section.

The abrupt change in failure stress at 70,000 psi is apparently caused by a rapid propagation of the region under residual compressive stress. Such an abrupt propagation of the plastic boundary has been found by Allen and Southwell on an analytical basis (18). Their theoretical study showed an abrupt propagation of the plastic zone in case of a sharply notched specimen when increasing the nominal stress from 63 to 64 per cent of the yield strength, and similar values for a specimen with a semicircular notch.

The above suggested mechanism is also supported by the appearence of the fatigue fractures of the specimens prestressed to various degrees. The specimens with no prestress show the transverse fatigue fracture commonly observed. With increasing prestrain the surface becomes curved with an increasing tendency to form a cone. A change in the shape of the fracture surface occurs at a nominal prestress of 70,000 psi. The specimen that was prestressed to 71,500 psi exhibited a transverse fracture surface similar to the specimens that were not prestrained. With further increased prestrain, the fracture surface becomes increasingly curved until a perfect cone results at a prestress of 97,240 psi. In other words the trend exhibited in the prestress range from 0 to 70,000 psi is repeating itself for prestresses larger than 70,000 psi.

The effect of short-time static tensile and compressive prestress at 300 F on the failure stress of the notched specimens of 2024-T4 aluminum alloy at the same temperature is also shown in Fig. 8. The behavior of this material is similar to that of 7075-T6 aluminum alloy. However, no abrupt change of the failure stress occurs as was observed with 7075-T6. Because of the large reduction of the failure stress by compressive prestrain, the starting stress for 2024-T4 alloy had to be lowered from 12,000 to 7000 psi. Tests with unstrained specimens started at the two different stresses resulted in the same failure stress. This indicates that coaxing effects are apparently small for this material and the particular test conditions.

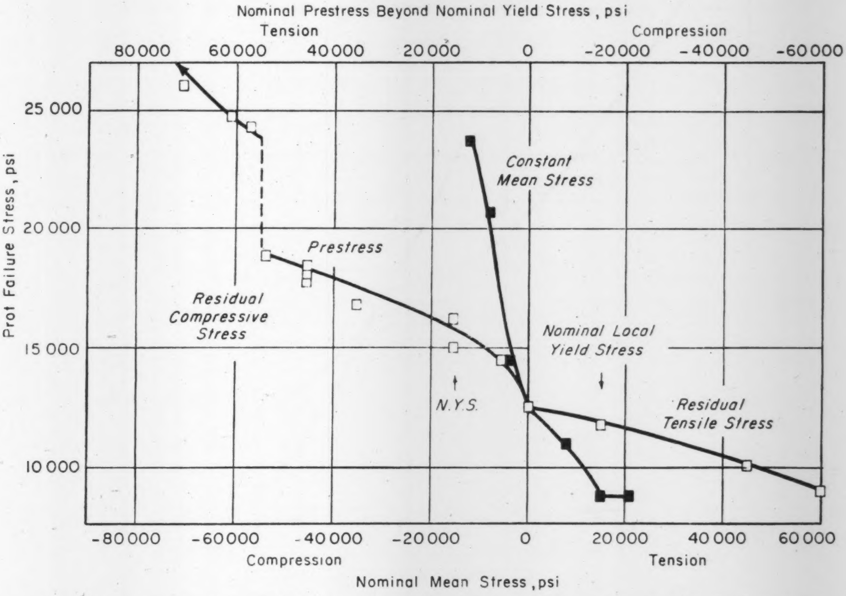


FIG. 9.—Comparison of the Effects of Static Mean Stress with Those of Residual Stresses Produced by Prestressing of Notched Specimens on the Fatigue Failure Stress of 7075-T6 Aluminum Alloy at Room Temperature.

N.Y.S. = Nominal yield stress.

Several tests on notched specimens were performed to compare the effect of residual stresses produced by prestressing with that caused by a constant static mean stress during a progressive load fatigue test. Such "stress range diagrams" are shown in Figs. 9 and 10 together with other curves for the alloys 7075-T6 and 2024-T4. Both diagrams indicate the trends characteristic of those observed in the customary constant stress amplitude tests. Comparative tests with the 7075-T6 alloy showed coincidence of the curve of the stress range diagram resulting from progressive load

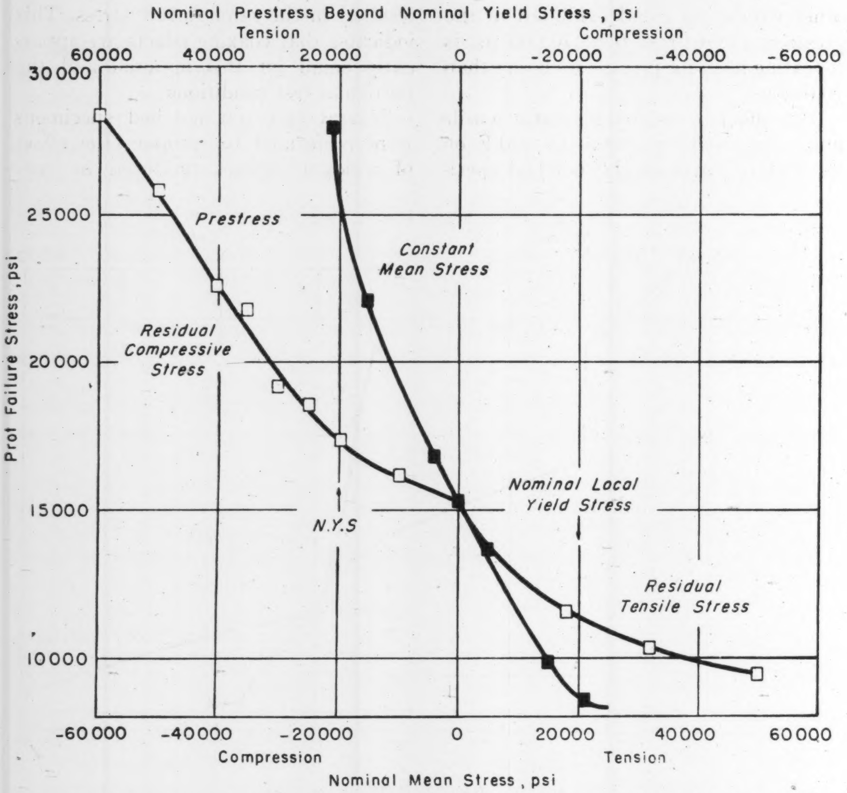


FIG. 10.—Comparison of the Effects of Static Mean Stress with Those of Residual Stresses Produced by Prestressing of Notched Specimens on the Fatigue Failure Stress of 2024-T4 Aluminum Alloy at 300 F.

N.Y.S. = Nominal yield stress.

fatigue tests with that from standard fatigue tests for 10^5 cycles to fracture.

Plotted in the same diagrams are curves for failure stress *versus* initial residual stress. The initial residual stress is plotted on the nominal stress axis. It was approximately determined from the difference between the nominal prestress and the nominal yield stress. The nominal yield stress was considered to be the maximum prestress up to which no effect of prestress on the fatigue failure stress occurs (compare with Fig. 8).

Comparison of the curves for the residual stress and constant mean stress indicates that relaxation of the residual stress is considerable. The relaxation times under the particular test conditions are, however, large enough even at 300 F in the case of 2024-T4, to cause an effect of residual stress on the fatigue behavior.

EFFECT OF PRIOR CREEP ON THE FATIGUE PROPERTIES OF NOTCHED SPECIMENS

Diagrams showing the effect of prior tensile creep stress on the fatigue properties of notched specimens of the alloys

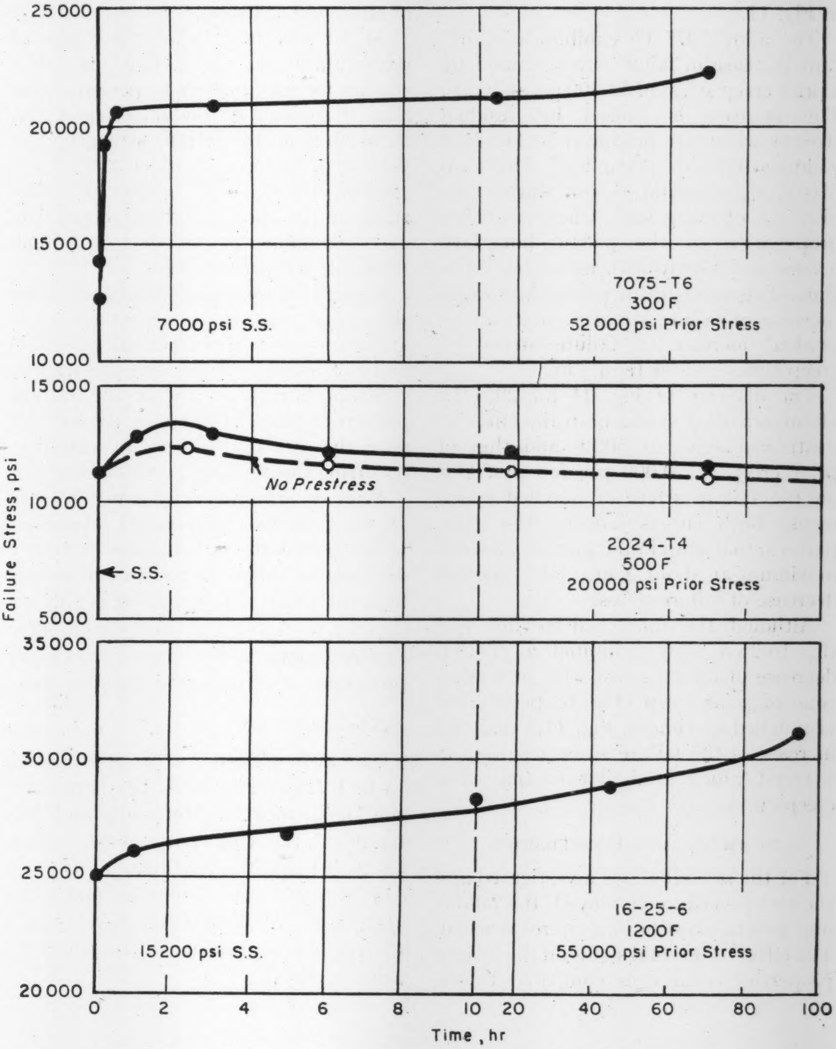


FIG. 11.—Effect of Creep Prestrain on the Fatigue Failure Stress of Notched Specimens ($K_t = 3.4$). S.S. = Starting stress.

7075-T6, 2024-T4, and 16-25-6 are shown in Fig. 11.

The alloy 7075-T6 exhibits a significant increase in failure stress caused by a prior creep stress of 52,000 psi at 300 F. The increase is caused by residual stresses which are produced by localized yielding under the static load. This local plastic deformation occurs during application of creep load, whereas further propagation of the plastic boundary occurs very slowly with increasing creep time. This mechanism results in a rapid increase of failure stress at first, and a gradual increase of failure stress for creep times greater than $\frac{1}{2}$ hr.

The diagram of Fig. 11 for 2024-T4 aluminum alloy shows both the effect of unstressed aging at 500 F and that of prior creep at 20,000 psi and at 500 F on the failure stress of notched specimens. Both curves indicate the same pattern, that is an initial increase, a weak maximum at about 2 hr, and a gradual decrease of failure stress.

Although the unnotched specimens of the 16-25-6 alloy exhibited a gradual decrease of failure stress with increasing time of prior creep (Fig. 6) prior creep of notched specimens, Fig. 11, caused an increase of the failure stress resulting in a trend which is similar to that of a creep curve.

SUMMARY AND CONCLUSIONS

For the several alloys investigated and the test procedure employed, the following general conclusions were reached. The effect of residual stress on the fatigue properties is large as compared to the effects caused by metallurgical reactions or strained hardening.

At temperatures below the range of recrystallization, relaxation is slow enough for many alloys to permit retention of significant residual stresses and their effect on the fatigue strength. The volume of material in which the critical residual stress occurs seems to affect significantly the relaxation mechanism and thus influences the effect of residual stress on fatigue behavior.

Because of the significant effect of residual stresses, the notch sensitivity is apparently decreased by tensile prestrain and increased by compressive prestrain.

Strain hardening affects the fatigue properties only in a minor degree, apparently due to recovery or softening occurring during fatigue stress history.

Prior deformation may cause reversal of precipitation in preaged materials, which results in softening and also in a decrease in fatigue strength for fatigue lives for which renewed aging is still not significant. Aging reactions which occur during fatigue and which affect fatigue properties are accelerated by prestrain.

Acknowledgments:

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A MECHANISM FOR NONPROPAGATING FATIGUE CRACKS*

By L. F. Coffin, Jr.[1]

Synopsis

Recent investigations on fatigue of metals have revealed the presence of nonpropagating fatigue cracks formed at the base of sharp notches under cyclic stress conditions. It has similarly been observed that a sharp notch is more severe a stress raiser in fatigue than a fatigue crack. A mechanism is proposed to explain these observations whereby the stress and strain in the base of the notch are fully reversed under the action of an external cyclic load but where for a crack, full reversal does not occur because of closure of the crack and reduction of the accompanying stress concentrating effects. The cyclic plastic strain, rather than stress, is used to compare conditions in notches and cracks. The mechanism predicts properly the effect of a superimposed mean stress on the propagation or nonpropagation of fatigue cracks in notched bars.

One of the interesting observations reported in the study of fatigue phenomena is that fatigue cracks formed under certain conditions of stress and geometry will not propagate. Fenner, Owen, and Phillips (**1**)[2] in 1951 discovered the existence of nonpropagating fatigue cracks in V-notch specimens of 0.1 per cent carbon mild steel. When the notch was of the order of 0.2 in. deep, with a 55 deg included angle and a root radius of 0.002 in., a nominal stress endurance limit of 8960 psi was obtained. However, test specimens run at stress levels of from 3800 psi to 4500 psi in all cases developed cracks at the root of the notch which did not propagate.

Frost (**2**) found that nonpropagating fatigue cracks occurred in reversed direct stress tests on an aluminum alloy of similar dimensions as those referred to above, in the range of from 2250 psi to 5600 psi alternating stress. In addition, several test specimens were run at 4450 psi for various numbers of cycles of stress. It was found that cracks appeared very early in the course of the cycling process and progressed to some depth but would not progress further. Figure 1 shows these results. It is evident from these observations that the fatigue crack is formed very quickly and then lies dormant. Frost found that the depth of propagation of the crack coincided with the position in the notch where the theoretically computed elastic stress (neglecting the crack stress concentration effect) equaled the fatigue limit of the material.

A corollary to the above observation is the fact that materials containing fatigue cracks produced by prior stress

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[2] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 575.

cycling have a greater resistance to subsequent cyclic stress than those containing sharp notches (3). In summarizing some work of Frost, Phillips (4) reported that for three materials (0.15 per cent carbon steel, a nickel-chromium steel, and an aluminum alloy) a notch root radius existed that would produce a minimum in the fatigue strength (the nominal stress to produce failure in 30×10^6 cycles). This critical radius appears to be around 0.0055 in. while above and below this radius the fatigue strength is greater. Table I shows the comparative results obtained for these three materials on an unnotched bar, a bar with a 55 deg V notch having a 0.002 in. root radius, and bars containing fatigue cracks which have been produced from prior fatigue testing of unnotched bars.

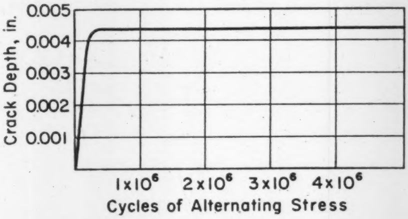

FIG. 1.—Course of Non-Propagating Fatigue Crack in Aluminum Alloy—After Frost (2).

TABLE I.—FATIGUE STRENGTH FOR VARIOUS MATERIALS AND STRESS RAISERS.

Type of Specimen	Stress for 50×10^6 cycles, psi		
	Mild Steel	Nickel-Chromium Steel	Aluminum Alloy
Unnotched	31 400	81 800	22 000
55 deg V notch 0.2 in. deep, 0.002 in. radius	8 750	10 100	5 600
Crack (from prior stress cycling of unnotched bars)	12 320	29 100	13 450

The phenomenon of nonpropagating fatigue cracks has aroused much discussion and a variety of mechanisms have been proposed to explain it. However, none of these explanations appear to the author to be entirely satisfactory. There is, however, a rather straight-forward explanation, described herein, which fits the observed facts.

AN EXPLANATION OF NONPROPAGATING FATIGUE CRACKS

In order to explain the occurrence of nonpropagating fatigue cracks, it is first necessary to consider the behavior of two stress quantities: (1) the stress to start a crack at the root of a notch and (2) the stress to propagate a crack in the region beyond the point where the notch geometry has any influence on the state of stress (designated 1 and 2 in Fig. 2). These two stress quantities are nominal stresses (load divided by original cross-sectional area) and are considered as separate functions of the initial notch radius. In Fig. 2, the behavior of these two stress quantities *versus* the notch radius is shown. Note that the stress (1) is shown to increase with notch radius. This is because with increasing notch radius the elastically calculated stress is reduced at the notch root. On the other hand, the stress (2) decreases with radius. Since nominal stresses are used rather than actual stresses, by the time the

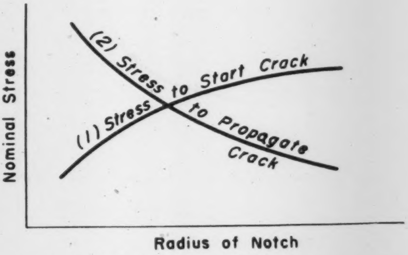

FIG. 2.—Variation of Nominal Stress with Notch Radius.

crack is beyond the influence of the highly stressed region produced by the notch geometry, the actual stress is above the nominally computed stress by the ratio of original area to actual area. Under the assumption that the notch radius is the only variable being changed, as it is decreased, the crack must grow to a relatively smaller percentage of the over-all cross-section before it propagates without the influence of the notch. Correspondingly then, the nominal stress decreases as the notch root radius increases. If the notch is very shallow, or the plate or bar very large, the stress (2) is a very flat function of the radius.

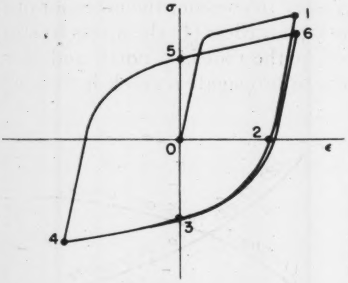

FIG. 3.—Stress-Strain Behavior at Notch Root.

In the representation given in Fig. 2, if curves (1) and (2) cross, then it is seen that below a certain notch root radius cracks will be formed which will not propagate. As yet no explanation has been given as to why these curves should cross and this remains to be done. The following argument can be used. Consider first a very sharp notch which (*a*) has not yet produced a fatigue crack, and (*b*) has developed a crack which has advanced beyond the influence of the stresses caused by the notch geometry. For both cases it is necessary to consider the state of stress and strain existing in the region where the crack will appear as in case (*a*), or where the next increment of crack will occur, as in case (*b*).

Case (*a*).—The stress-strain behavior in the highly stressed region of case (*a*) is demonstrated in Fig. 3. One may then trace through the above curve as follows: starting at point *0* a tensile stress is applied which produces an elastic and then plastic strain to reach point *1*. This plastic behavior occurs only in the small region around the notch root; elsewhere the material is elastic. Removal of the applied load causes the local stress first to decrease to point *2* and then to go into compression, reaching some point *3* when the load is fully released. It is assumed here that the large area of elastic material will return the strain in the plastic region to zero, resulting in a residual compressive stress. The application of an external compressive load results in a continuation of the hysteresis loop producing a compressive strain and further compressive stress to point *4*. Unloading returns the strain to zero again and the stress to a tensile value at *5*. Further cycling of the external load between tension and compression then leads to a cyclic strain between point *6* in tension and point *4* in compression until a fatigue crack develops. The mechanism whereby the fatigue crack occurs is not known; however it is known that the number of

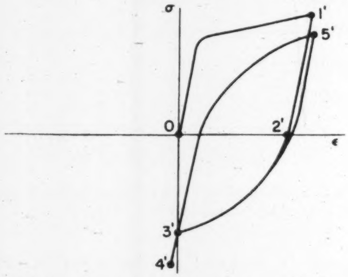

FIG. 4.—Stress-Strain Behavior Beyond Notch Root.

cycles to produce failure by fatigue varies inversely with the magnitude of the cyclic plastic strain (5).

Case (*b*).—We now consider the stress-strain behavior when the crack has progressed through the region of the notch to that part of the specimen uninfluenced by the original geometry (Fig. 4). First application of a tensile load produced the curve *0-1′* as before. Here the strain developed at point *1′* is somewhat greater than at point *1* in the previous example, but since the sharpness of the notch, in case (*a*), is assumed to approach that of the crack, in case (*b*), the difference in strain is assumed to be relatively small. Release of the load results in the curve *1′-2′-3′*, following the same argument as above. However, with application of an external compressive load, the situation is now quite different. This is because the crack is now closed up completely when the strain returns to its zero value. Thus the crack can now transmit a compressive stress, but without the stress concentrating effects of case (*a*). Hence the stress-strain curve in the plastic region ahead of the crack due to the applied compressive load is given by curve *3′-4′*. There is no plastic behavior accompanying this application of load because the stress concentration effects have been removed by the closing of the crack. Thus, when the compressive load is returned to zero, the strain returns to *3′*. A subsequent tensile load results in a stress-strain curve going to point *5′*, and continued cycling establishes the hysteresis loop *5′-3′-4′-3′-5′*.

Comparing now the amount of cyclic plastic strain which is produced in the hysteresis loops of case (*a*) and case (*b*), if the strains at *1* and at *1′* are of the same magnitude, then the plastic strain range for the notch is greater than that for the crack, and consequently causes a lower resistance to fatigue. Under these circumstances, a value for external cyclic stress can be found where a crack starts and grows in the region of the notch but will stop after progressing beyond the influence of the notch. On the other hand, if $(\epsilon_{0\text{-}1'})_p$ is greater than $2 \times (\epsilon_{0\text{-}1})_p$, that is, where the notch plastic strain is low in relation to the plastic strain at the tip of the crack, the crack will continue to propagate at the same rate as it did within the region influenced by the notch.

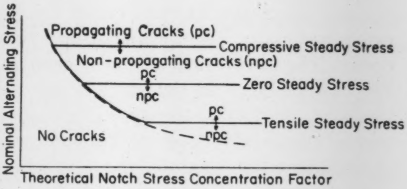

FIG. 5.—Effect of Mean Stress on Non-Propagating Fatigue Cracks—After Frost and Dugdale (7).

The argument presented here to explain nonpropagating fatigue cracks is that when the crack which has formed at the notch root grows sufficiently deep, the crack can close during external compression without producing a stress concentrating effect. It must be necessary for the crack to have advanced sufficiently far to be out of the influence of the notch where a stress concentration can occur in compression. Thus, nonpropagating cracks should grow as far as the edge of the locally stressed region around the notch, but no farther. This conforms with the observations above.

Recent test results of Frost and Dugdale (7) are of interest in the present discussion. They investigated the effect of superimposed static stress on the propagation of fatigue cracks in notched bars. It is observed that with increasing steady tension superimposed upon the cyclic stress, nonpropagating cracks are observed only if the notch is made increasingly sharp (the notch radius is less). On the other hand, in the presence of a superimposed steady compressive stress, nonpropagating cracks are found for notches that are less sharp than in the case where no mean stress is present. This can be represented in Fig. 5 where the theoretical notch stress concentration factor is plotted as the abscissa and the nominal alternating stress is plotted as the ordinate. The dashed line represents the boundary between the regions where no cracks are formed and the region where the cracks, either of the propagating or nonpropagating type occur. The theoretical stress concentration factor here is determined on the basis of elasticity theory. The region where cracks do occur is further subdivided into two sections, namely, where they do not propagate (npc), and where they do (pc). The point of particular interest here is that in the presence of a mean or steady compressive stress, the boundary between propagating and nonpropagating cracks is raised so as to require a large cyclic stress before the cracks that are found will propagate. On the other hand, when a mean tensile stress is present, the boundary is lowered below that for a zero mean stress, such that it becomes relatively easy for cracks to propagate, once formed. When a given alternating stress is applied, cracks will apparently occur, by the above representation, if a notch of some critical radius is present. The superposition of a steady stress increases or decreases the likelihood that the crack will propagate depending on whether the superimposed steady stress is tension or compression.

It is of interest to interpret these results in the light of the mechanism presented here, namely, that nonpropagating cracks are due to the ability of the crack to transmit compression without stress concentration. First it seems reasonable to assume that the presence of a mean stress would not affect the conditions necessary to initiate a crack in the notch since the cyclic plastic strain would not be altered significantly. The effect of a mean compressive stress is such as to keep the crack closed over a greater portion of the cycle. The magnitude of the cyclic plastic strain is thus reduced because of the decrease in the stress and strain concentrating effects of the closed crack during cycling. On the other hand, the effect of a mean tensile stress is to open up the crack during a greater portion of the cycle, to increase the magnitude of the cyclic plastic strain, and to extend the fatigue crack. Thus, there appears to be good qualitative agreement between the most recent results of Phillips and the mechanism proposed here for explaining nonpropagating fatigue cracks.

DISCUSSION

In the light of the above reported observations and the mechanism proposed to explain these observations, it might be interesting to speculate as to the effect that residual stresses play on the fatigue resistance of materials. First, it is well known that fatigue is a highly selective, surface sensitive property. Thus, the nature of the surface, including its method of preparation, its roughness, and the environment to which it is exposed, are significant contributing factors in determining fatigue life. It is well known that fatigue cracks start at surfaces (6) except under unusual situations where the stress state is such as to produce the maximum shearing stress well beneath the surface, as in rolling contact. At the surface, the formation of cracks is highly selective, and even the smallest defect can act as a source for crack initiation. It is not unreasonable to assume that these defects may not be unlike the sharp notches discussed above

As in the above discussion, some of these defects may lead to propagating fatigue cracks, whereas others will produce nonpropagating cracks. This suggests, then, that the steady stresses that are acting in the material, whether they are produced by internal or external means, will have the principal effect of causing cracks already formed at defects to propagate or not, depending upon the magnitude and the sign of these stresses.

From the above point of view, the effect of a surface residual stress, for example, is not to prevent the occurrence of fatigue cracks but rather to alter the conditions which cause these fatigue cracks to propagate. It would then be expected that surfaces that are extremely smooth and have comparatively few defects will be quite insensitive to the effects of residual stress, unless any tensile stress is completely obliterated by a very high residual compressive stress. On the other hand, surfaces that are comparatively rough would be highly susceptible to the effect of residual stress, since a large spectrum of defects would exist. The presence of a surface compressive residual stress would prevent the propagation of many of the cracks which might have occurred otherwise, while a surface tensile residual stress will enhance the propagation of these cracks, once they have been formed.

The depth of the residual stress layer is also of significance. When the surface is very rough, the effective or highly stressed region ahead of the notches produced by these rough surfaces may be quite deep, and the presence of the compressive residual stress may not be beneficial if the depth of this layer is less than the effective depth of the notch. From the point of view taken here, then, it would seem that not only are the magnitude and sign of the residual stress important factors, but of equal importance is the depth of the residual stress layer.

From the reasoning given above, it would seem possible by the proper selection of the magnitude and depth of the residual stress layer to achieve the same fatigue strength for a rough, poor-quality surface as it would for a surface with the best polished finish. By introducing a residual stress layer at the surface, a barrier is introduced to inhibit the propagation of fatigue cracks formed by surface defects. Provided this barrier is of sufficient depth and magnitude, the externally applied stress required to cause the crack to break through this barrier would be the same regardless of the initial surface conditions. This point should be verified experimentally.

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A FATIGUE FAILURE HYPOTHESIS BASED UPON STABILIZED UNIDIRECTIONAL SLIP*

By D. G. Younger, Jr.[1]

Synopsis

The total slip process taking place during the cyclic stressing of metals is viewed to be that of a to-and-fro deformation superimposed upon a unidirectional deformation. The manifestation of these deformations, however, is not necessarily macroscopic in character. They are microscopic in origin and, in general, may only be revealed through powerful magnification as a slip phenomenon within the microstructure. From the evidence presented, the unidirectional slip process is known to cease very early in the fatigue life, but not until after considerable fragmentation and disorientation of the grains has produced a stabilized pattern of striations. The alternating slip, on the other hand, continues throughout the fatigue life, intensifying the striations until cracks are developed and propagated to failure. This two-fold slip phenomenon forms the basis of a failure hypothesis in which the early stabilization of unidirectional slip is considered to play a major role. Recent observations in conjunction with historical studies are shown to lend support to the failure mechanism proposed. A test of the hypothesis using direct stress *S-N* data for a number of ferrous and nonferrous alloys shows very good correlation.

In recent years the mechanism producing fatigue failure has been sufficiently clarified to indicate that fatigue strength determinations and lifetime predictions cannot be based on static material properties. This of course, has been received with some reluctance since the advantages of establishing such a simple relationship could hardly be overestimated. With the hope that some relatively simple relationship may still be found, attention has now focused on the dynamic material properties as a measure of fatigue.

The purpose of this paper is to review briefly the findings presented by a number of investigators and to attempt to integrate these into a fatigue failure hypothesis. The studies considered most relevant are those which have directed attention to the deformations attendant to cyclic stressing. Particular emphasis is placed on those findings which have indicated the existence of a stabilized level of unidirectional slip.

Statement of the Hypothesis

The evidence that has slowly unfolded during the past century supports the contention that considerable unidirectional

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slip takes place during the early stage of cyclic stressing of metals. This unidirectional slip process produces severe crystal disorientations, fragmentations, and striations that form into a stabilized pattern early in the fatigue life. The end result of this phenomenon is very similar in nature to the stabilized yielding condition observed macroscopically[2] on a number of metals by several investigators (**1,2,3, 4,5**).[3] In general, however, the yielding (or unidirectional slip) process is microscopic in origin and it does not necessarily reveal its presence, for all metals, at the macroscopic level of observation. The degree to which microscopic processes are reflected onto macroscopic readings is dependent to a large extent on the strain-hardening characteristics of the metal studied.

The mild steels examined at the turn of the century by Bairstow (**1**), and more recently by Lidstrom and Lazan (**4**) showed uninhibited straining characteristics. That is, the microscopic yielding mechanism was not obscured from macroscopic observation by the strain-hardening phenomena. Mild steels of this type have often been referred to as "strain-softening metals," since they display a decreasing dynamic modulus[4] during the early stages of fatigue life before the stable condition is reached.

When these materials undergo cyclic axial stress, a saturation level of unidirectional slip is found which exhibits the characteristic of being a single-valued function of the maximum stress. The locus of stress levels *versus* saturation strains is referred to here as the "stabilized locus curve." This saturation phenomenon is viewed to be commonplace for all metals at the microstructural level, and it is with this viewpoint in mind that the following statement of the hypothesis is advanced:

During the cyclic stressing of ductile polycrystalline metals a locus of stress levels *versus* saturated unidirectional deformations exists. The points forming the locus are single-valued functions of the maximum stress. The cyclic life for a given range of stress is uniquely determined by the corresponding range of stabilized deformation as measured from the locus.

A corollary to this hypothesis is: Any ductile polycrystalline metal will exhibit equal cyclic life for equal ranges of stabilized deformation as measured from the locus.

DETAILED EVIDENCE SUPPORTING THE STABILIZED LOCUS CURVE

Observations by Bauschinger and Bairstow:

About a century ago strain as well as stress during fatigue began to be studied in detail. It was then that Bauschinger (**6**) realized the need of obtaining more information in fatigue studies than merely the number of cycles to fracture. He demonstrated that the statically determined limits of proportionality can be greatly modified by certain cyclical variations in stress. This phenomenon is now well known as the "Bauschinger effect." For completely reversed stress, Bauschinger found that the limits of proportionality adjusted to stabilized magnitudes and constituted a fixed range between the tensile and compressive limits. He then presented a theory that this stabilized range of stress could be indefinitely endured. For completely reversed stress, Bauschinger called the upper limit of this "safe" range the "natural elastic limit."

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[2] Macroscopic behavior and phenomenological behavior are considered as phenomena that can be observed by large-scale or extensometer-type measurements.

[3] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 593.

[4] The term "dynamic modulus" has found extensive use in the work of Lazan and associates. This term is synonymous with the "cyclic secant modulus." It is defined as the slope of the diagonal line of the stress-strain hysteresis loop.

The general truth of Bauschinger's theory was demonstrated some thirty years later by the classical experiments of Bairstow (1) made at the National Physical Laboratory. Using an axle steel, Swedish iron, and a Bessemer steel, Bairstow made a very comprehensive study of the hysteresis loops and permanent set commonly found to exist during cyclic loadings. His experiments are well known and have been discussed in much of the present-day literature. However, it is doubtful if the full significance of these early observations have been realized.

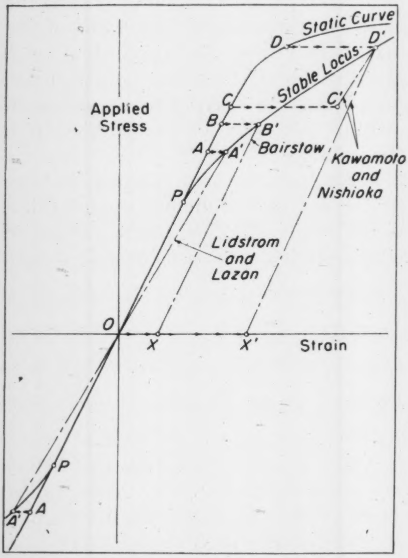

FIG. 1.—Unidirectional Strains Observed During Cyclic Stressing.

The general observations made by Bairstow are depicted in Fig. 1. Also included in this figure are the findings of other experimenters to be discussed later. For purposes of illustration, the "static curve" in the figure can be assumed to be that obtained by Bairstow in an ordinary static tension test. Under a repeated axial stress equal to, say *OB*, the first cycle did not show a measurable yielding of the test specimen, but repeated application of this stress produced considerable yielding. After a relatively small number of cycles the deformation at the maximum stress level had moved to some position, *B'*. At this point the yielding ceased, and thousands of additional cycles failed to extend the unidirectional deformation.

In a similar manner the yielding represented by *AA'* and *DD'* were obtained by repetitions of stress *OA* and *OD*, respectively. For the repeated stress *OP* there was no yielding of the specimen. In fact, the stress level *P* was considered by Bairstow to represent Bauschinger's natural elastic limit. In other words, the stress range *OP* was found to be one half the safe range of stress for cycles of completely reversed stress.

Bairstow found that the stabilized points *PA'B'D'* seemed to join smoothly with the static curve as shown in the figure. He expressed the opinion that extensions such as *BB'* would probably be caused even by an alternating stress which would not produce final failure. The region between the static curve and the stabilized locus curve *OPA'B'D'* appeared, therefore, to be one which could be explored only by cyclic stressing. A single application of stress, as in a static test, could give no information about this region.

Observations by Lidstrom and Lazan:

Recently, Lidstrom and Lazan (4) performed experiments that have shed additional light on the progress of macroscopic deformations in strain-softening metals. In some respects their measurements on a mild steel paralleled rather closely those made by Bairstow. However, these more recent studies employed completely reversed stresses rather than repeated stresses. Lidstrom and Lazan directed attention to those maximum stress levels which were both near, and considerably above, the reversed stress-endurance limit. They, too, placed emphasis on the existence of a stabilized locus curve relating maximum stress and deformation during axial loading.

Figure 1 can be used in discussing their findings on the 1020 steel which are deemed applicable to this paper. Although the "static curve" shown is not representative of that found for the mild steel, it will suffice as the starting line for indicating the cyclical yielding phenomenon. When a typical axial stress *OA* above the endurance limit was completely reversed for 10^3 cycles, the yielding of the test specimen reached the point *A′* in both tension and compression as indicated. Then the test specimen was subjected to a very severe stress history. This envolved the application of a number of cycles having decreasing stress amplitude followed by cycles of increasing stress amplitude. After this intermediate loading procedure, the original stress level, *A*, was reached once more. Finally, at the original stress level a total of 10^4 cycles was accumulated on the test specimen. It was noted that no significant yielding beyond *A′* had occurred even though an additional 9000 cycles had been applied.

Using separate test specimens, the same procedure of loading was applied at a number of other initial stress levels. The findings were unchanged. The unidirectional slip process had apparently stabilized during the first 1000 cycles and no additional yielding at the maximum stress level could be developed during the next 9000 cycles. A line joining the stabilized points formed a smooth curve, of the type *PA′B′D′*, and essentially reconfirmed the findings of Bairstow discussed above. It is also significant to note that Lidstrom and Lazan found no yielding at the stress level represented by *P*, and for the axial loading the reversed stress *P* was found to be the endurance limit for the mild steel. The similarity of this "safe" stress with Bauschinger's natural elastic limit is of major interest.

Observations by Kawamoto and Nishioka:

The cyclic stress experiments performed by Kawamoto and Nishioka (7) on a mild steel, a high-carbon steel, and a spring steel are very significant. They have not only added more evidence in support of the stabilized locus curve, but also have added another important finding. Their tests were performed in torsion, thus introducing a stress gradient effect; however, the deformations observed are similar in nature to those noted in the direct stress tests. The Japanese investigators found that the same locus curve was established by the stabilized yielding phenomenon regardless of the mean stress. They used both repeated stresses and alternating stresses of high mean stress, and the same locus curve was produced. The essentials of their findings are shown in Fig. 1. The yielding, *DD′*, at the maximum stress was found to be the same whether the stress cycle on the specimen was repeated between *O* and *D* or alternated between *C* and *D*. In either case, the yielding was found to stabilize at *D′* early in the fatigue life. The two ranges of stress, *OD* and *CD*, were found to produce radically different fatigue lives even though the same amount of yielding at the maximum stress level had been experienced. This means that the magnitude of the yielding at the maximum stress is a function only of that stress level and is unaffected by the range of stress and the eventual fatigue life.

Observations Concerning the Dynamic Proportional Limit:

The term "dynamic proportional limit" has been defined and studied on the phenomenological scale by a number of investigators. In Fig. 1, the stress level corresponding to the prevailing definition of the dynamic proportional limit is *P*. The experiments by Gough (8), Smith and Wedgwood (3), Lazan and Wu (9), and Demer (10) have shown that the dynamic proportional limit provides a good approximation to the fatigue endurance limit for reversed stress and lies well within the fatigue scatter band for many metals.

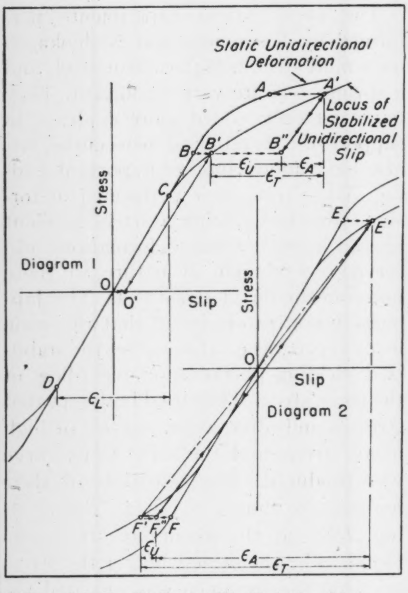

FIG. 2.—Cyclic Stress-Slip Relationships.

There are other metals, however, that do not exhibit, on the phenomenological scale, this correlation between the dynamic proportional limit and the endurance limit. The light alloys, for instance, studied by Demer (10) and Blatherwick and Lazan (5), show no correlation of this type. These metals severely strain-harden, and the true dynamic proportional limit is obscured from macroscopic observation. Only a pseudo limit of proportionality is observed, and this is usually found to be considerably higher in stress than the endurance limit. For some metals then, the true dynamic proportional limit represented by *P* in Fig. 1 may be revealed only through powerful magnification of the internal slip processes.

DETAILED EVIDENCE SUPPORTING THE FAILURE HYPOTHESIS

The deformation studies made by the investigators mentioned earlier, as well as those by the author (11), during cyclic loading of metals have prompted the formulation of the hypothesis stated at the beginning of this paper. To facilitate a discussion of the failure mechanism inherent in the hypothesis, consider the first diagram in Fig. 2. The upper curve represents a typical virgin stress-strain curve which should be considered as nothing more than a static relationship. The lower curve, on the other hand, is a locus curve of dynamically stabilized unidirectional slip. The shape of this curve cannot be duplicated by any known means of unidirectional loading as in the static test. It is not a curve segment of a hysteresis loop. In general, this locus curve can only be found by the loading procedures discussed earlier in conjunction with Fig. 1. The locus curve shown in Fig. 2 represents the locus of maximum stresses and stabilized slips theorized to exist at the microstructural level for all ductile polycrystalline metals. However, as an expedient to this discussion, the curves selected for this figure are typical of those found on the phenomenological scale by the investigators mentioned earlier.

Deformations During Alternating Stress:

The combined findings of Bairstow, Lidstrom and Lazan, and Kawamoto and Nishioka, discussed previously, have shown the locus of stabilized deformations to be a function only of the superior (maximum) stress and not the range of stress. Therefore, if the stress range from A to B, in Fig. 2, is selected for convenience to be a range for cyclic stressing, then the strain at the superior stress, A, will translate through unidirectional slip to the stabilized position A'. Simultaneously, the strain at point B will move through position B' and on to some position B''. The strain at the minimum stress level, B, does not stop at the locus curve position, B', because the elastic unloading from the maximum stress forces the lower point to continue on to B''. The over-all movement of A to A' and B to B'' is the result of a unidirectional deformation occurring during the to-and-fro deformation. After A' and B'' have been reached, the cycling continues between these points during the greater part of the fatigue life until failure.

The Damage Process:

The findings of Kawamoto and Nishioka showed that ranges of stress having a common maximum stress would develop the same amount of yielding at the maximum stress level. This, of course, emphasized that the yielding at the maximum stress is a function only of that stress level and is unaffected by the magnitude of the minimum stress. Evidently this phenomenon adds considerable light to the mechanism contributing to fatigue failure. If the magnitude of the yielding occurring at the maximum stress is independent of the range of stress, it must also be independent of the severity of the damage process. Conversely, the damage process must be independent of the yielding taking place at the maximum stress.

The existence of such a nondamaging slip mechanism was indicated some years ago by the metallographic observations of Gough and Hanson (**2**) and more recently by Hunter and Fricke (**12**). The latter authors associated the initial slip process with a decrease of high internal energy culminating in a state of equilibrium. As a result of these findings, the yielding that takes place at any stress level in passing from the static state (static curve) to the stabilized state (locus curve) is considered to have little or no effect on the fatigue life.

Once the yielding at the maximum stress level has ceased, the two real contributors to fatigue damage can be brought into focus. One of these is the cyclical permanent set.[5] During repetitions of the unloading half of the cycle, a system of residual stresses is developed which introduces a cyclical permanent set in the material. The magnitude of this set is a function of both the maximum stress and the range of stress. For example, as shown in Fig. 1, repeated stressing between either O and B or O and D produces not only the yielding at the maximum stress but also the cyclical permanent sets OX and OX', respectively. In Fig. 2, the cyclical permanent set developed in cycling between stress levels A and B is labeled ϵ_U. On the microstructural level, the permanent set has been shown to be accompanied by a disorientation of the crystals, and a stabilized pattern of striations (**13,14, 15,16**) is developed.

The second mechanism producing damage is that of the alternating strain taking place between the two stress levels.

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[5] The cyclical permanent set is defined as the irrecoverable strain measured between the stabilized locus curve and the lower extreme of the dynamic modulus.

The alternating strain is a damage process that continues on to failure after all other strains of a unidirectional nature have ceased. In Fig 2, ϵ_A represents the magnitude of the alternating strain taking place between stress levels *A* and *B*. Wood (16) has referred to this type of straining as a pure fatigue process where elementary translations through relatively few lattice spacings are intensifying. The crucial feature of pure fatigue is that it proceeds to intensify those striations that were previously developed during the unidirectional slip process. The summation of these two straining processes (unidirectional plus alternating) is shown in the figure to be ϵ_T. This constitutes the total strain mechanism affecting the fatigue life.

Deformations During Reversed Stress Near the Endurance Limit:

Now consider cycling between stress levels *C* and *D*. This constitutes a complete reversal of stress. As shown in Fig. 2, no unidirectional slip movement at *C* and *D* would be experienced. These particular points lie on the stabilized locus curve at the outset. In fact, cycling between *C* and *D* would be expected to show a very long, if not indefinite, cyclical life since these points correspond to the dynamic proportional limit. For this range of stress the total strain mechanism is shown to be composed entirely of the cyclical (pure fatigue) component. The existence of an indefinitely long life for these stress limits would indicate that the magnitude of the total slip mechanism or a rate of damage is just within the limits that can be healed by fluctuations of thermal energy (14) or other processes within the structural arrangement. In other words, a type of damaging and healing mechanism must be in balance. The limiting total strain that enables such a balance to occur is labeled ϵ_L in the figure. All ranges of stress exhibiting this magnitude of the total strain mechanism will receive equal benefit from the healing process and the same indefinitely long fatigue life will ensue. The more the total strain, ϵ_T, exceeds the limiting value, ϵ_L, the greater will be the out-of-balance between the damage and healing mechanisms and the shorter will be the fatigue life. In general, those ranges of stress showing equal total strain, ϵ_T, will show equal fatigue life.

Deformations During Reversed Stress Above the Endurance Limit:

In considering completely reversed stresses exceeding the dynamic proportional limit, attention should be focused on the second diagram in Fig. 2. Cycling between stress limits *E* and *F* will produce the same type of eventual straining action already discussed. However, the sequence of events may differ somewhat at first.

As cycling between *E* and *F* begins, the usual slips will take place and it will be found that point *E* moves toward E' and point *F* toward F'—each extreme stress level adjusting toward the stabilized state of strain. Ordinarily during this movement, a hysteresis loop joining E' and F' is developed which has the dynamic modulus $E'F'$. After a number of cycles, some materials may show a reversal in the movement at the minimum stress level when observed from the phenomenological point of view. This possibility is shown as movement F' to F''. Such a reversal ordinarily becomes apparent as a measurable increase in the dynamic modulus. If the apparent reversal in slip direction does occur it may be associated with a form of delayed strain hardening (5) or, possibly, differences in the influence of compression and tension on the slip phenomena. Nevertheless, the total strain, ϵ_T, affecting the fatigue life is, as before, the summation of the two strain components, ϵ_U and ϵ_A.

Dynamic Modulus Variations:

Dynamic modulus variations of the type referred to in the above discussion were measured by Lidstrom and Lazan (4). Their measurements were made on test specimens of mild steel subjected to completely reversed axial loading. Results typical of those presented in their work are shown in Fig. 3. The dynamic modulus was observed to decrease very rapidly during the first 1 per cent of the fatigue life. This was then followed by a gradual increase in modulus beginning long before failure. The slight modulus increase was explained in another study (5) to be the consequence of a delayed strain-hardening phenomenon. The magnitude of these dynamic modulus changes were verified by other work by Lazan (17) and Lazan and Wu (9).

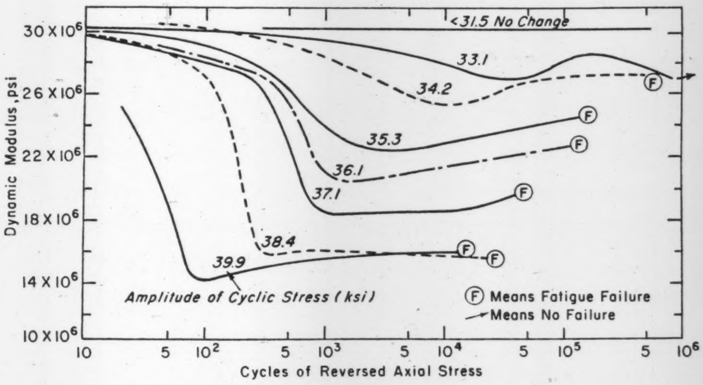

FIG. 3.—Dynamic Modulus Variations During Cyclic Stressing of Mild Steel.

It should be noted from Fig. 3 that no modulus changes were experienced for reversed stresses less than 31.5 ksi. This stress level corresponded to the dynamic proportional limit and was very near the endurance limit for the mild steel. Note, also, the rather erratic variation of the dynamic modulus occurring at the low-stress levels and the crossover of the variations at the two highest stress levels. These observations tend to support the contention that once the minimum value of the dynamic modulus has been established, further variations in the modulus will have little or no effect on the eventual fatigue life during direct stressing. This, of course, infers that the basic array of crystal disorientation and striations is developed when the dynamic modulus reaches a minimum and further cycling only intensifies the slipbands which have been formed. As a result of this intensifying process, some modifications of the strain-hardening (16) may take place, resulting in a modulus change, but this probably is no more than a secondary effect.

Internal Stresses Evaluated by Magnetic Analysis:

About ten years ago a series of interesting tests was performed by Duke (18) using a method of magnetic analysis. He employed the principles of magnetostriction of ferromagnetic materials to evaluate internal stresses during fatigue action. The method is based on the fact that a distortion of the crystal lattice in a specimen, whether induced by a static or dynamic loading, will have a measurable effect on the magnetic properties. For those unfamiliar with the physics of the method, the work of Cavanagh and Wlodek (19) is suggested. Typical results obtained by Duke are shown in Fig. 4. Duke explained that the mild steel tested was initially possessed with the usual internal stresses resulting from manufacture. The specimens were cycled at 120 cps in reversed bending above the endurance limit. At the end of each 25,000-cycle period, the specimens were removed from the fatigue tester and the magnetic changes were noted.

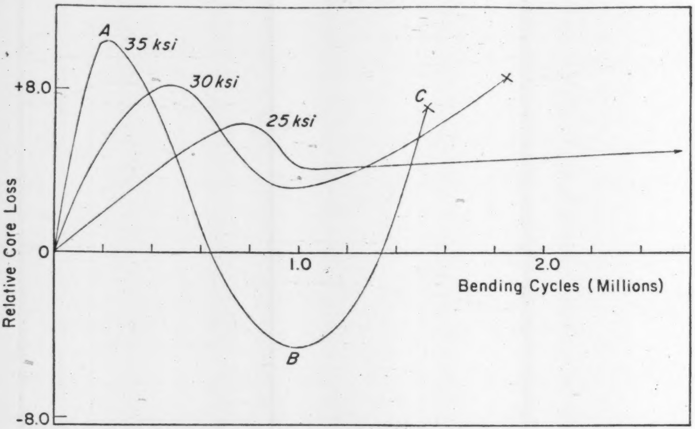

FIG. 4.—Typical Cyclograph Study on Mild Steel.

After the initial periods of cyclic loading, measurements showed that the internal stresses were relieved by a slip mechanism considered to be nondamaging in nature. This process of internal adjustment, which also accomplishes a state of magnetic neutrality, is represented by path *OA* in Fig. 4. It is believed that this initial nondamaging adjustment corresponds to the movement *EE′* and *FF′* shown earlier in Fig. 2.

Between points *A* and *B* in Fig. 4, according to Duke, the initial nondamaging adjustment process is replaced by a semi-elastic, semi-plastic straining process. At the lowermost point *B*, the material reached a limiting condition of plastic movement. At the limiting plastic strain position, *B*, it was noted that intensified microscopic stress fields had been developed. This straining mechanism between *A* and *B* appears to be closely associated with the establishment and intensification of an array of crystal disorientations and striations resulting from the combined unidirectional and alternating strain process.

When the plastic straining reached the limiting magnitude at position *B*, another mechanism came into focus. Between *B* and *C* it was found that the energy which had previously produced plastic flow was now directed to lattice destruction. Duke found the intensity of the destruction to be entirely dependent on the applied stress, once the specimen reached position *B*, the limit of plastic strain. This final mechanism probably consists of the crack-propagation phase, which is relatively slow in the presence of stress-gradient effects common to bending specimens.

The three stress levels shown in Fig. 4 refer to the reversed limits assigned to each series of tests. Each curve portrays the phases of straining just discussed. The 25-ksi curve depicts conditions existing near the endurance limit. At this stress level, Duke believed that the damage-healing process was in near balance. Stresses at, or below, the endurance limit did not produce the lattice destruction phase.

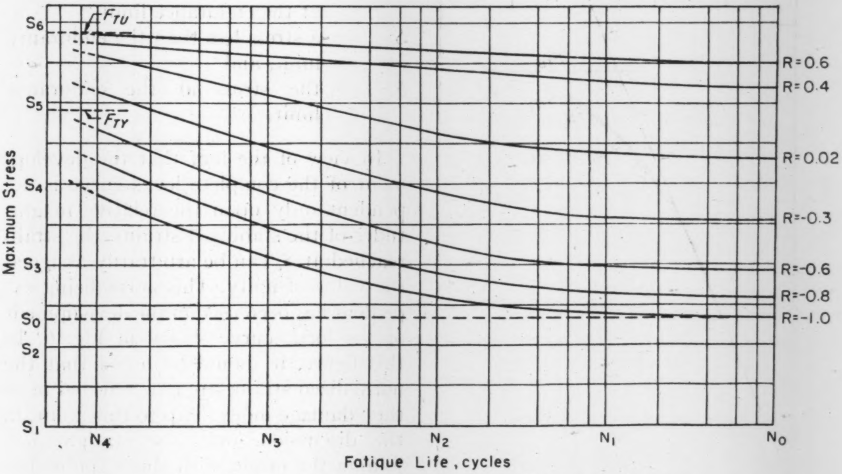

FIG. 5.—Typical *S-N* Fatigue Curves for Constant Stress Ratios.

A TEST OF THE HYPOTHESIS

As stated in the hypothesis, "the cyclic life for a given range of stress is uniquely determined by the corresponding range of stabilized deformation as measured from the locus." For this to be true for a given material, all of the *S-N* data for a number of mean stresses must establish a single stress-strain relationship similar to the locus curve referred to in Figs. 1 and 2. A procedure that shows the existence of such a singular relationship for fatigue data was recently developed in a preliminary study (20) based upon the assumption that the failure of test specimens follows the "limiting strain" hypothesis presented here. The fatigue data for a number of metals were studied and the correlation was found to to be very good. The procedures used and the correlations found are discussed in the following sections of this paper.

Development of the Locus Curve from Typical S-N Curves:

Consider fatigue test specimens tested under direct stress varying sinusoidally between a maximum stress, S_{max}, and a minimum stress, S_{min}. It is known that the test frequency has negligible effect on the number of cycles which cause failure, at least up to the highest frequencies of normal engineering interest (9,21). For the stress range, $S_{max} - S_{min}$, let the number of cycles causing failure be N.

A typical set of *S-N* curves obtained under direct stress is shown in Fig. 5. These curves are representative of those generally faired through the failure test

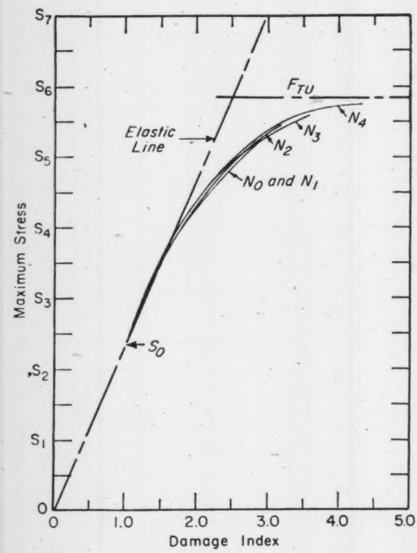

FIG. 6.—Development of Locus Curve.

points. The values, R, indicate the ratio of the minimum stress to the maximum stress applied during the constant stress cycle. The largest value of N covered by the data is shown to be N_o. For actual fatigue data, the magnitude of N_o generally ranges between 10^7 and 5×10^8 cycles. From the lower curve, $R = -1$, the reversed stress giving a lifetime of N_o cycles is found to be S_o. The very prolonged lifetime associated with S_o indicates that the damage-healing process is in near balance and this stress level can be considered as the accepted value of the endurance limit. From the findings of the investigators mentioned earlier, the material undergoing this reversed stress remains in a cyclic state that is very nearly elastic in nature. That is, the stress range S_o to $-S_o$ corresponds to the range C to D in Fig. 2. This range of stress determines a length of the locus curve which can be considered as linear. Algebraically, this linear portion of the locus curve can be expressed as,

$$\gamma(S) = \frac{S}{S_o}\gamma(S_o) \qquad \text{..........(1)}$$

where:

$\gamma(S)$ = the stabilized strain for stresses less than the endurance limit,

$\gamma(S_0)$ = the stabilized strain for a stress at the endurance limit,

S = a stress less than the endurance limit, and

S_o = the stress at the endurance limit.

In view of the fact that the development of the complete locus curve is dependent only upon the relative magnitudes of the stabilized strains, the strain reached at S_o can be arbitrarily assigned the value of unity. This normalizing expedient has been used in the development of the locus curve shown in Fig. 6. In this figure, it should be noted that the normalized strains are now referred to as the "damage index." Up to this point, in the discussion, only the straight line joining the origin with the S_o point has been established.

To continue the development of the locus curve beyond S_o, a range of stress must now be selected from Fig. 5 which has a mean stress greater than zero and which still produces a fatigue life of N_o. From the $R = -0.8$ curve, for instance, determine the values of S_{max} and S_{min} corresponding to the cyclic life N_o. The value of the damage index, $\gamma(S)$, corresponding to S_{min} can be read[6] from the

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[6] In evaluating the damage index for compressive stresses, it will be assumed that the locus curve is antisymmetrical about the origin. Therefore, for simplicity, only the tensile half of the curve need be shown as long as a negative sign is assigned to the compressive stresses.

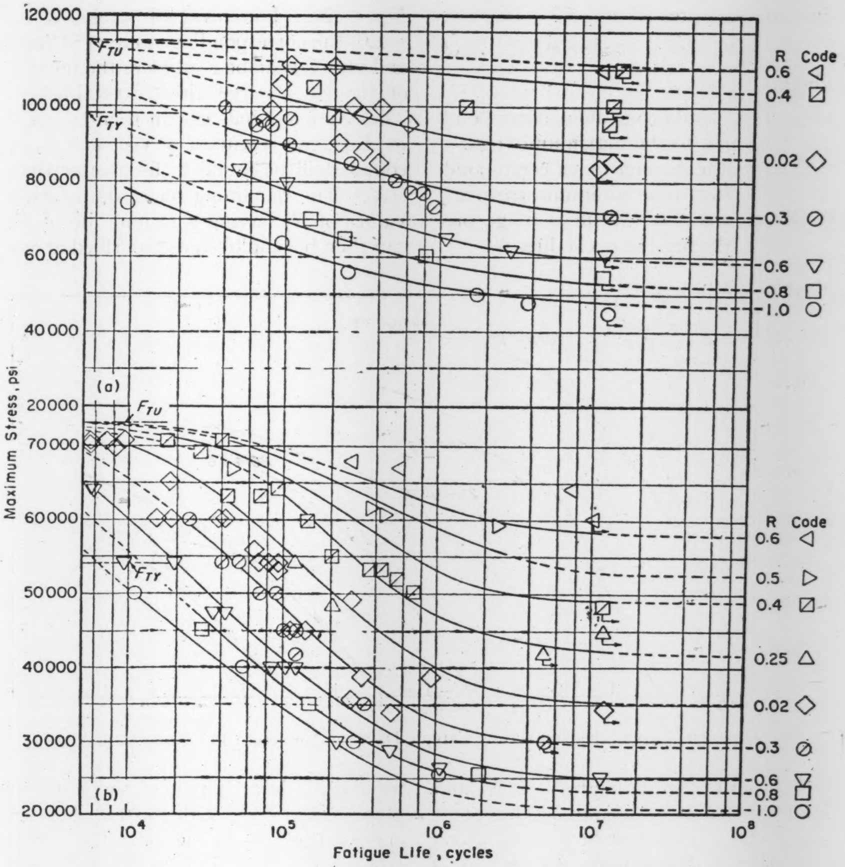

FIG. 7.—Fatigue Curves for (*a*) Normalized SAE 4130 Steel and (*b*) 2024-T3 Aluminum Alloy.

linear portion of the locus curve already established. Now, by referring to Fig. 2, it can be seen that the limiting total strain, ϵ_L, that is commensurate with the endurance limit is equivalent to twice the damage index given for S_o in Fig. 6. The normalized value of the limiting total strain, therefore, is found to be equal to two units of damage. According to the hypothesis, this total strain damage not only produces a cyclic life, N_o, during reversed stress, but it also produces a cyclic life, N_o, for all levels of mean stress. To obtain the value of the damage index corresponding to the S_{max} under consideration, it is only necessary to add two units to the damage index, $\gamma(S_{min})$, previously determined. Algebraically, this can be expressed as:

$$\gamma(S_{\max}) = \gamma(S_{\min}) + \delta(N) \ldots\ldots (2)$$

where:

$\gamma(S_{\max})$ = the damage index corresponding to the maximum stress,

$\gamma(S_{\min})$ = the damage index corresponding to the minimum stress, and

$\delta(N)$ = the total strain damage producing the cyclic life, N.

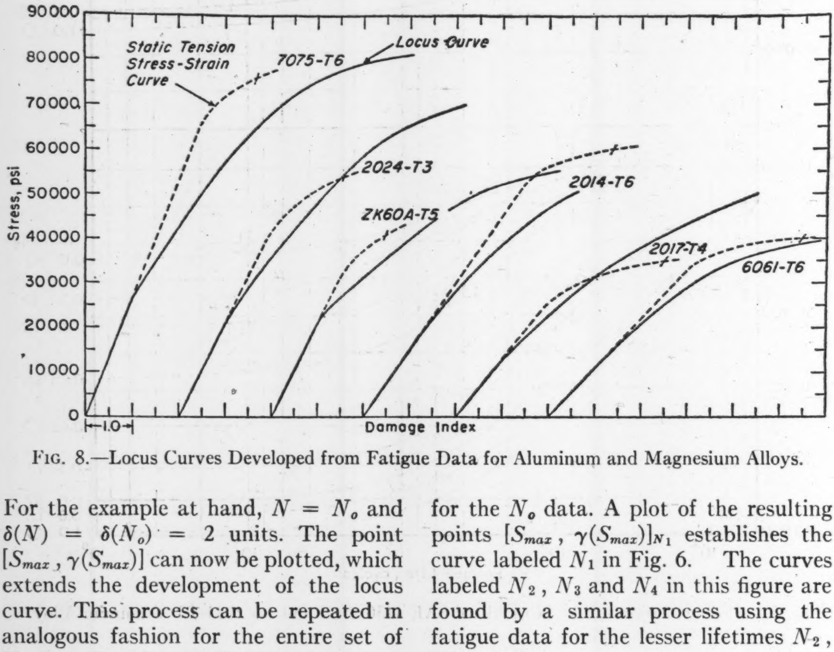

FIG. 8.—Locus Curves Developed from Fatigue Data for Aluminum and Magnesium Alloys.

For the example at hand, $N = N_o$ and $\delta(N) = \delta(N_o) = 2$ units. The point $[S_{max}, \gamma(S_{max})]$ can now be plotted, which extends the development of the locus curve. This process can be repeated in analogous fashion for the entire set of N_o data. In other words, the process can be repeated for all of the stress ranges covered by the data which exhibit a lifetime of N_o cycles. Referring to Fig. 5, this would entail using data for $R = -0.6, -0.3, 0.02, 0.4$ and 0.6 in a manner similar to that used for $R = -0.8$. Then, by joining all of the resulting points, $[S_{max}, \gamma(S_{max})]_{N_o}$, the curve labeled N_o in Fig. 6 is established.

Now consider the data for fatigue life $N_1 < N_o$. For reversed stress, $R = -1.0$, the corresponding value of $\gamma(S_{\min})$ and $\gamma(S_{\max})$ can be read from the portion of the locus curve already established. These damage indices can then be used[7] in Eq 2 to evaluate $\delta(N_1)$. $\delta(N_1)$, of course, will be found to be greater than $\delta(N_o)$. The remaining set of data (other values of R) having a fatigue life of N_1 can then be employed as described above for the N_o data. A plot of the resulting points $[S_{max}, \gamma(S_{max})]_{N_1}$ establishes the curve labeled N_1 in Fig. 6. The curves labeled N_2, N_3 and N_4 in this figure are found by a similar process using the fatigue data for the lesser lifetimes N_2, N_3 and N_4.

It should be noted that in the process of locus curve development, the locus is assumed straight only below the endurance limit, S_o. For absolute values of S_{min} greater than S_o, the extrapolation of the locus curve is always based upon a previously established value of $\gamma(S_{min})$. That is, the locus curve development is

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[7] The damage index is a negative value if obtained for a compressive stress.

made through a process of overlapping curve segments with the lower extremity of each curve segment coinciding with that portion of the locus already established. As shown in Fig. 6, the curve segments overlap for a greater portion of their length, thus describing essentially the same path.

Correlation with Actual Fatigue Data:

Fatigue test data for several metals were located through the literature. The data selected met the requirement of being obtained under direct stress for a number of mean stresses. The metals and source references are as follows:

(a) Aluminum alloy 2014-T6.......... (22)
(b) Aluminum alloy 2017-T4.......... (23)
(c) Aluminum alloy 2024-T3.......... (21)
(d) Aluminum alloy 6061-T6.......... (22)
(e) Aluminum alloy 7075-T6.......... (21)
(f) Magnesium alloy ZK60A-T5........ (24)
(g) Normalized SAE 4130 steel........ (21)
(h) SAE 4340 steel.................. (25)

The *S-N* fatigue failure data for the normalized SAE 4130 steel are shown in Fig. 7(*a*). It should be of interest to note that the typical *S-N* curves presented in Fig. 5, for purposes of discussing the locus curve development, are actually duplications of the curves appearing in Fig. 7. The dashed portion of these curves indicate the regions in which predictions are made beyond the existing data points. The locus curve for the normalized SAE 4130 steel is shown in Fig. 9. The fact that a locus curve can be found which in turn predicts with reasonable accuracy the failure of specimens at various levels of mean stress and alternating stress adds emphasis to the compatibility of the proposed failure mechanism.

The *S-N* data for the 2024-T3 aluminum alloy are shown in Fig. 7(*b*). Again, the curves drawn through the data are those predicted by the proposed failure law. Despite the large scatter exhibited by this set of data, the correlation is still reasonable. The single locus curve which is commensurable with these *S-N* curves is shown in Fig. 8.

The dashed curves shown in Figs. 8 and 9 are the virgin tension stress-strain curves which have been normalized and presented for purposes of comparison. The short slash mark has the usual meaning of the 0.2 per cent offset yield strength commonly used for materials not exhibiting a definite yield point.

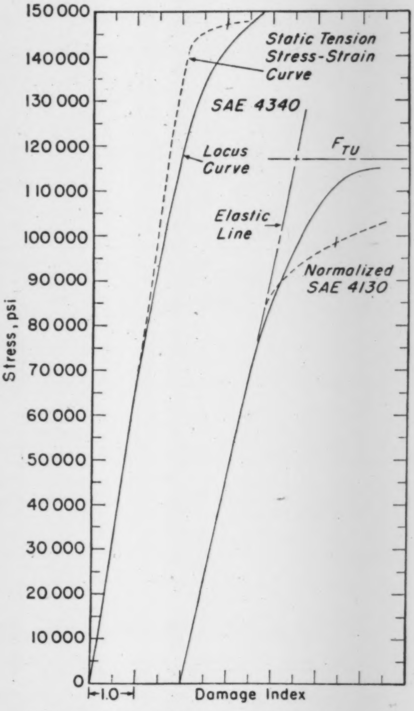

FIG. 9.—Locus Curves Developed from Fatigue Data for Alloy Steels.

The *S-N* data and the predicted fatigue curves for 7075-T6 aluminum alloy, ZK60A-T5 magnesium alloy, and SAE 4340 steel are shows in Figs. 10(*a*), 10(*b*), and 10(*c*) respectively. The cor-

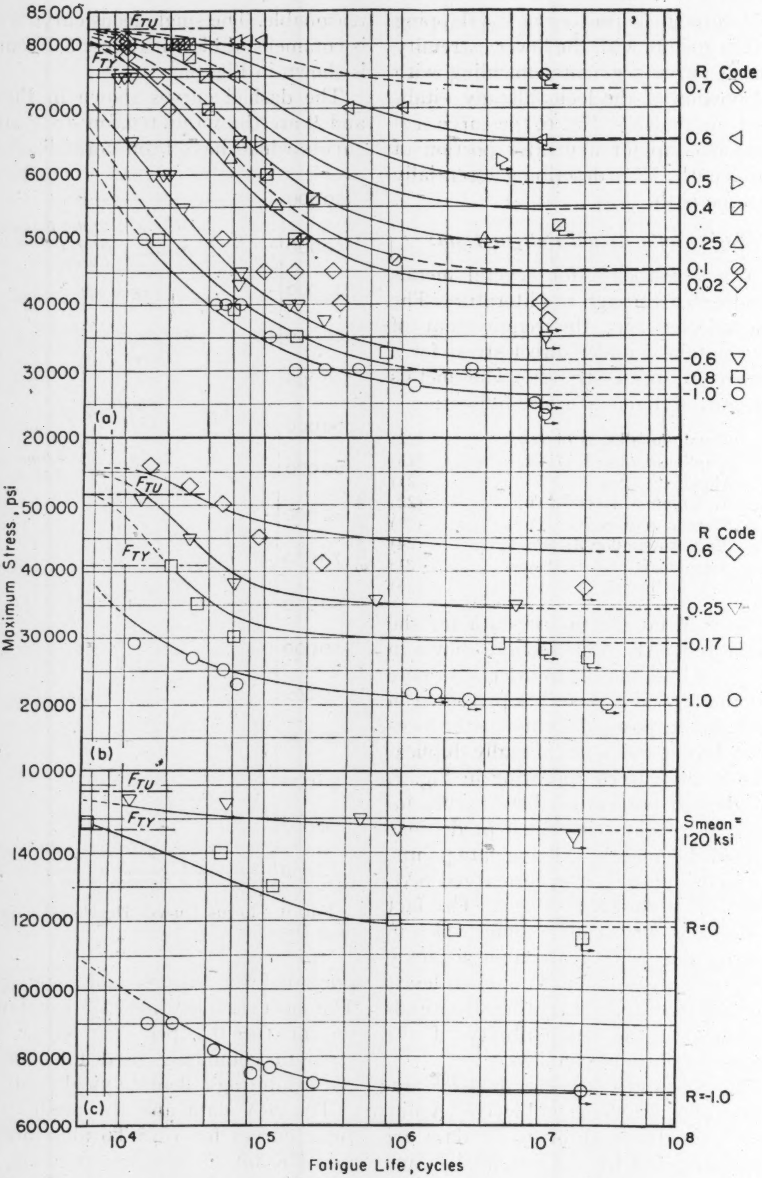

FIG. 10.—Fatigue Curves for (*a*) 7075-T6 Aluminum Alloy (*b*) ZK 60A-T5 Magnesium Alloy and (*c*) SAE 4340 Steel.

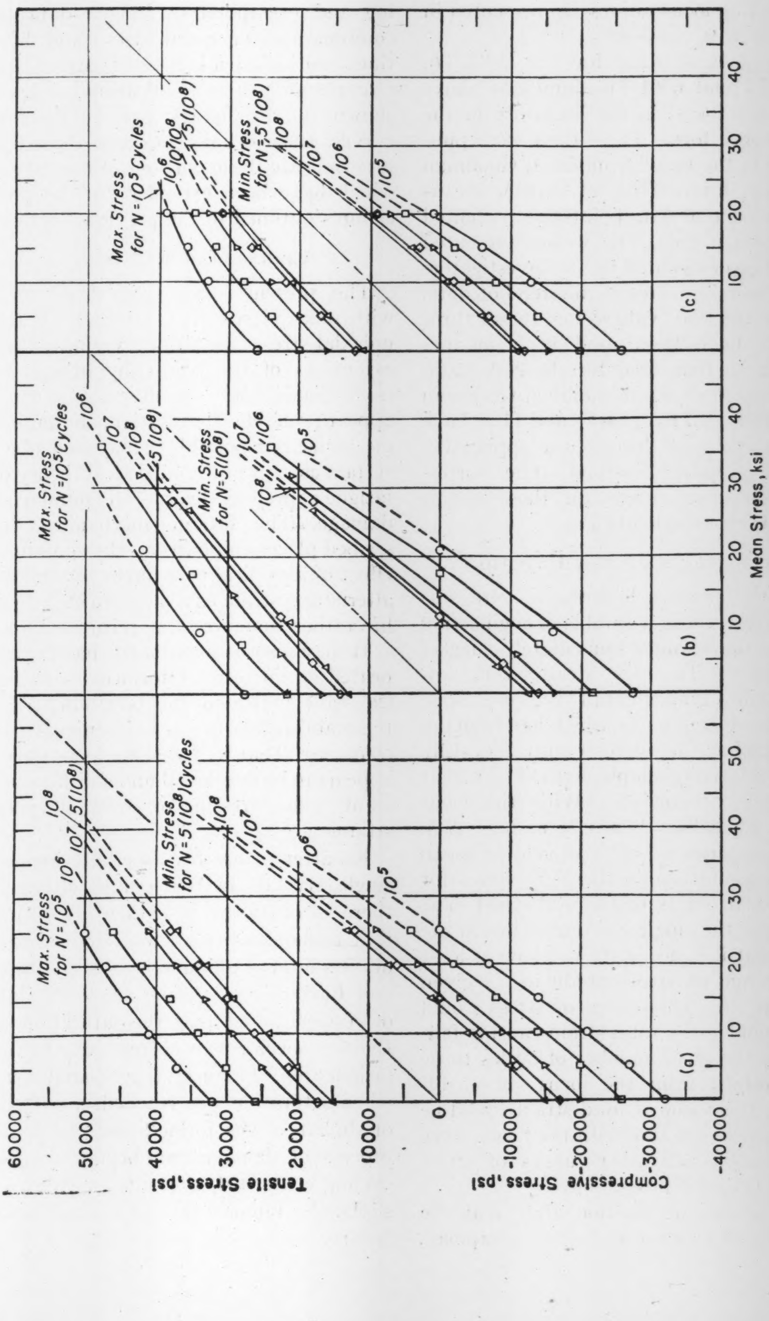

FIG. 11.—Predicted Fatigue Diagrams for (*a*) 2014-T6 Aluminum Alloy (*b*) 2017-T4 Aluminum Alloy and (*c*) 6061-T6 Aluminum Alloy.

responding locus curves are presented in Figs. 8 or 9.

The fatigue data for the 2014-T6, 2017-T4 and 6061-T6 aluminum alloys were not found in the literature in the *S-N* curve form. These data were published in the familiar modified Goodman diagram form. This, of course, means that no actual data points are presented for these metals. The values presented have been estimated by the investigators to be representative of the trend of data. The estimated values for these three metals have been used in a manner similar to that used for the *S-N* data. The curves shown in modified Goodman form in Figs. 11(*a*), 11(*b*), and 11(*c*) have been developed through the application of the proposed method. The corresponding locus curves for these metals have been included in Fig. 8.

APPLICATIONS OF THE HYPOTHESIS

If the present hypothesis were sufficiently verified, a number of engineering applications would immediately suggest themselves. The most ideal of these applications would entail the use of a stabilized locus curve which had been determined at the microstructural level by some relatively simple method, say that of magnetic analysis. With the locus curve available, then only a single *S-N* curve determined at a convenient mean stress would enable the *S-N* curves for any mean stress to be established. The need for the single *S-N* curve lies in the fact that it is necessary to know at least one range of stress producing a given fatigue life. This range of stress would then enable the total strain causing failure in the given number of cycles to be determined from the locus curve. Finally, this value of total strain would be used in conjunction with the locus curve to establish all other ranges of stress exhibiting this fatigue life.

A second application that may be worthy of mention is that of interpolating and extrapolating fatigue data to combinations of mean stress and lifetimes not included in a test program. An example of such an application has been shown in the figures containing *S-N* curves. Dashed-lines shown in these figures indicate those portions of the curves which have been extended by the procedures outlined in the previous section.

CONCLUDING REMARKS

The present study has dealt only with uniaxial stressing. However, if the uniaxial stress case can be verified, then extensions of the failure hypothesis to cover biaxial and triaxial stressing cases appear probable. In addition, the failure mechanism proposed here may well affect certain aspects of the statistical theory of fatigue as well as theories on cumulative damage. The fatigue mechanism described places emphasis on the combined effect of a cyclical permanent set and an alternating strain as a measure of fatigue life rather than stress amplitudes alone.

It may also be mentioned that the repeated strain tests performed by Bailey (26) have indicated the possibility that the stabilized locus curve is universal in character. That is, the same locus curve appears to be developed under either constant cyclic stressing or constant cyclic straining.

No attempt has been made to include single crystals in this study, although there is considerable evidence showing that these also reach a well-defined saturation limit of unidirectional slip. Ebner and Backofen (27), using single crystals of copper, found that the saturation of the test specimens occurred early in the fatigue life. Further, they found that when saturation was reached, a pattern of slipbands was formed and the basic process of damage had begun. As the cycling continued, the initial pattern of slipbands intensified and spread until failure.

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DISCUSSION

MR. JO DEAN MORROW.[1]—This paper has been stimulating, but at the same time, a bit confusing. Some of the confusion stems from the fact that the author has used a combined graphic and analytical method for determining a fatigue damage index curve for material tested at various mean stresses. Further confusion arises when an attempt is made to justify the existence of such a damage index curve on the basis of a hodgepodge of information gathered from the many references. The entire spectrum of knowledge in the field of fatigue is used, from metallographic observations of slip, to cyclic stress-strain behavior, to experimentally determined *S-N* curves. Yet no clear picture emerges of precisely what the justification is for assuming fatigue damage to be related to stabilized unidirectional slip.

The data used in this paper is limited to R $\geqslant -1.0$. Would this not also be a limitation on the "hypothesis" since the same fatigue behavior would be predicted for compressive mean stresses as for tensile mean stresses? It is well known that this is not so.

It would seem that the author's contribution is mainly that of furnishing the designer with a method of plotting mean stress fatigue data from minimal information (that is, a damage index curve and an *S-N* curve). The similarity of the damage index curve and the stabilized locus curve is certainly worth noting, but does not seem to me to be sufficient basis for the development of a fatigue failure theory.

MR. D. G. YOUNGER (*author's closure*).—As has been recognized by Mr. Morrow, the data collected for use in this paper have been limited to mean stresses equal to, or greater than, zero. The omission of data for the compressive mean stresses stems from the fact that much of the compression fatigue data appears to be clouded with a high degree of uncertainty. That is, either the method of loading the specimen has introduced eccentricities of unknown magnitude or the measurements have been inadequate for determining the actual stresses developed.

From the type of compression fatigue data that are available, it would be difficult to say whether or not the hypothesis is limited to mean stresses of zero or greater. It is evident from the literature that data of the stabilized locus curve variety are scarce, if not non-existent, for compressive loadings. Therefore, the assumption made in this paper that the "locus of stabilized unidirectional slip" is antisymmetrical with respect to the origin is admittedly open to argument. However, before one gathers momentum for such an argument it should be borne in mind that fatigue does not actually occur under compressive stress. The use of compressive mean stresses in discussing fatigue behavior only adds to the confusion. The principal question that needs an answer is, "What is the magnitude of the tensile mean stress introduced by the compressive mean loading during fatigue action?" The correct answer to this difficult question would shed considerable light on the factors governing the shape of the modified Goodman diagram in the region of compressive mean loads.

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SUMMARY OF PROCEEDINGS OF THE SYMPOSIUM ON BASIC MECHANISMS OF FATIGUE

This Symposium is somewhat of a departure from the general type of publication in that an attempt is made to determine what basically affects fatigue performance. Generally the interest has been concerned with standardizing test methods, and in applying results to actual design. The papers in this Symposium clarify some of the mechanisms leading to fatigue damage and help to emphasize the statistical nature of the material behavior during cyclic loadings.

The papers and discussions in this Symposium were presented at the Second Session of the Sixty-first Annual Meeting of the American Society for Testing Materials, held at Boston, Mass., June 22 to 27, 1958. It was sponsored by Committee E-9 on Fatigue. T. J. Dolan of the University of Illinois organized the Symposium and presided over the session.

The papers included in the Symposium are:

Introduction—T. J. Dolan
Progress Report on Dislocation Behavior in Lithium Fluoride Crystals During Cyclic Loading—R. E. Keith and J. J. Gilman
Fatigue Crack Formation in Silver Chloride—P. J. E. Forsyth
Internal Friction, Plastic Strain, and Fatigue in Metals and Semiconductors—Warren P. Mason
Slipband Formation and Fatigue Cracks Under Alternating Stress—M. R. Hempel
Cycle-Dependant Stress Relaxation—JoDean Morrow and G. M. Sinclair
Recent Observations on Fatigue Failure in Metals—W. A. Wood

The papers with discussions were issued as *ASTM Special Technical Publication No. 237* entitled "Symposium on Basic Mechanisms of Fatigue."

THE RESPONSE OF HIGH-STRENGTH STEELS IN THE RANGE OF 180,000 TO 300,000 psi TO HYDROGEN EMBRITTLEMENT FROM CADMIUM-ELECTROPLATING*

By E. P. Klier,[1] B. B. Muvdi,[2] and George Sachs[3]

Synopsis

The embrittlement of seven high-strength steels due to the action of hydrogen introduced by cadmium-electroplating has been studied in sustained-load tests. Strength levels from 180,000 to 300,000 psi as suitable for the various steels were examined for a variety of initial conditions of stress concentration. All steels were found to be embrittled in some measure after cadmium plating.

Failure promoted by cadmium plating is affected by the experimental conditions and has been discussed at length in the report. In the hydrogen-bearing zone a crack is initiated and then depending on the experimental conditions may propagate to failure of the cross-section through overloading. Crack development is apparently dependent, in part, on the composition and is minimized by reduction in carbon content or by an increase in silicon content.

The failure of high-strength steels through the action of electrodeposited hydrogen in the presence of a suitable stress system has been intensively investigated during the past five years. Thus, while hydrogen embrittlement is a very generally encountered phenomenon, it is now possible to describe characteristics of this effect which are perhaps best restricted to the consideration of high-strength steels. One of the most characteristic features of hydrogen induced failure in high-strength steels is the delay time effect for failure to take place under the action of statically applied loads. The first plausible explanation of this behavior was advanced by Petch and Stables (1)[4] who proposed a suitably modified form of the Griffith-Orowan (2) theory of static fatigue in glass. This theory requires the development of a suitable crack which through reduction in the cross-section leads to eventual overloading of the uncracked cross-section and thus to failure.

The crack formation proposed by Petch and Stables was experimentally verified by Raring and Rinebolt (3), Klier, Muvdi, and Sachs (4), and Barnett and Troiano (5).

Barnett and Troiano have investigated at length the development of the crack for specimens containing electrodeposited hydrogen and propose that failure takes place in three stages, namely,

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* Presented at the Sixty-first Annual Meeting of the Society, June 22–27, 1958.

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[4] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 619.

TABLE I.—CHEMICAL COMPOSITION OF STEELS.

Alloy	Size and Shape	Alloying Elements, per cent									
		Carbon	Manganese	Phos-phorus	Sulfur	Silicon	Nickel	Chromium	Molyb-denum	Vanadium	Copper
4340	4¼ in. round	0.41	0.79	0.013	0.016	0.31	1.83	0.77	0.23	..	..
4330 (Vanadium modified)	3½ in. round	0.32	0.82	0.010	0.021	0.28	1.82	0.89	0.41	0.07	..
98B40	4½ in. round	0.46	0.80	0.019	0.022	0.41	0.88	0.79	0.20	..	..
Tricent (Inco)	4¼ in. square	0.39	0.74	0.014	0.014	1.54	1.83	0.83	0.38	0.07	..
Crucible UHS-260	4 in. square	0.35	1.20	0.023	0.017	1.62	..	1.26	0.32	0.20	..
Hy-Tuf	4½ in. round	0.25	1.30	0.022	0.018	1.47	1.75	0.38	0.38	..	..
Super TM-2	3 in. round	0.41	0.72	0.012	0.014	0.61	2.98	1.15	0.44	..	0.14

TABLE II.—HEAT TREATMENTS FOR VARIOUS STEELS EXAMINED.

Alloy	Austenitizing Temperature, deg Fahr	Tempering Temperature; Strength Level, psi				
4340	1525	400 F (275 000)	500 F (270 000)	700 F (235 000)	800 F (215 000)	1000 F (165 000)
4330 (Vanadium modified)	1600	250 F (265 000)	500 F (235 000)	750 F (210 000)	1000 F (180 000)	
98B40	1550	400 F (300 000)	575 F (270 000)	700 F (230 000)	800 F (205 000)	900 F (195 000)
Tricent (Inco)	1600	400 F (295 000)	550 F (275 000)	700 F (270 000)		
Super Hy-Tuf	1700	500 F (270 000)	800 F			
Hy-Tuf	1575	700 F (230 000)	(250 000)			
Super TM-2	1600	500 F (275 000)				

1. *Crack Initiation.*—The crack develops at the base of the notch and passes rapidly through the hydrogen-rich surface layers.

2. *Crack Propagation.*—If the applied load is sufficiently low that fracture does not result during stage 1, further crack development is suppressed until hydrogen can diffuse to the crack front. Second stage crack growth is, therefore, controlled by the hydrogen diffusion rate.

3. *Fracture.*—If the crack is allowed to propagate indefinitely, the cross-section will be eventually reduced to the point where the fracture strength of the notch section is exceeded and fracturing in the normal way then takes place.

This description of the failure process while stated in greater detail establishes the role of hydrogen in leading to failure in much the same terms as proposed by other investigators.

The notch strength for a steel after tempering in the range to promote high-strength conditions is dependent on the specimen size (**6, 7, 8**). With increase in the specimen size, the notch strength is lowered. The notch strength still is much higher than that measured under sustained loads for hydrogen embrittled steel, however. As the specimen size continues to increase, it is probable that the notch strength asymptotically approaches a limiting value, and this corresponds to a limiting notch strength which has the characteristics of a material constant. From the experimental data so far obtained, it is proposed that the limiting notch strength lies above the notch strength observed in the small (0.3-in. diameter) sustained-load specimens.

In the sustained-load tests the effect of hydrogen is effectively to reduce the section and in this way to promote failure. If a larger specimen is employed, the surface to volume ratio will be reduced, the average hydrogen content of the specimen will be reduced, and the crack will penetrate a relatively shorter distance through the specimen. As a consequence, the notch strength of larger specimens electroplated should be relatively raised.

Important as is the establishment of the physical characteristics of the failure of high-strength steels through the action of hydrogen, a perhaps more pressing need is the elimination in these materials of the phenomenon as a source of structural failures. This, in principle, is possible either by suitable processing to counteract the effects of hydrogen embrittlement or by the use of materials that are impervious to fractures arising from the action of electrodeposited hydrogen. A usual processing treatment for the elimination of hydrogen embrittlement is a low temperature baking operation. This treatment is not, however, generally adequate. Practical difficulties arise in the retention of the required properties in the steels and in the electrodeposit, if complete elimination of harmful hydrogen is achieved. Principal emphasis in the present investigation has as a consequence been directed at the evaluation of available materials for susceptibility to hydrogen embrittlement as measured in sustained-load tests. The fracture surfaces obtained for the several types of specimens have been regularly examined and the characteristic types are discussed.

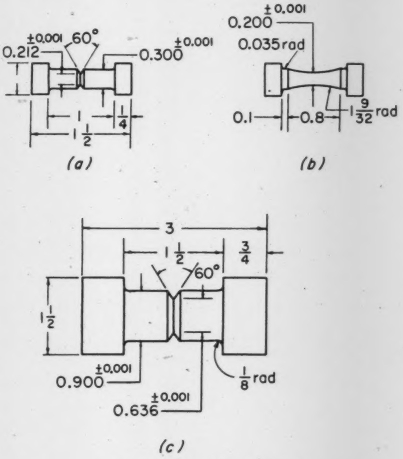

FIG. 1.—Test Specimens.

(*a*) 0.3 in. diameter stress-rupture notch specimen.

(*b*) 0.2 in. diameter stress-rupture smooth specimen.

(*c*) 0.9 in. diameter stress-rupture notched specimens.

EXPERIMENTAL PROCEDURE

Materials:

The compositions and finished form of the steels studied are given in Table I.

Heat-Treatment:

Specimens were austenitized in a suitable salt bath, oil-quenched and tempered for 4 hr in a forced-draft muffle furnace. The austenitizing and tempering treatments employed for the respective steels are given in Table II.

Test Specimens:

All specimens were rough machined to +0.015 in. on each surface prior to heat treatment. After heat treatment, specimens were machined to the dimensions given in Fig. 1.

TABLE III.—STRESS CONCENTRATION FACTORS AND CORRESPONDING NOTCH RADII FOR SUSTAINED-LOAD SPECIMENS.

Type	Unnotched Diameter, in.	Notch Diameter, in.	Stress Concentration Factor, K	Notch Root Radius, in.
Stress-Rupture specimens....	0.300	0.212	3	0.011
			5	0.0035
			10	0.0008

Smooth specimens were polished by the application of emery cloth under a suitable coolant. The direction of movement of the polishing cloth was along the axis of the specimen.

Notches were obtained by the use of suitably ground cutting tools. The dimensions of the notches as calculated by Peterson (9) from Neuber's theory are given in Table III.

Plating Conditions:

In an earlier work the embrittlement action of electrodeposited hydrogen arising from potential cleaning operations was discussed (4). In the present work hydrogen embrittlement due to hydrogen introduced into the steel during cadmium-electroplating is studied. The composition of the cadmium-plating bath was as follows:

Cadmium-Plating Solution:	
Cadmium Oxide..............	4 oz per gal
Sodium Cyanide..............	16 oz per gal

The bath was operated at room temperature at a current density of 200 ma per sq in. The plate thickness obtained was from 0.00025 to 0.0005 in. for smooth specimens. For the notched specimens, metallographic examination revealed that plating was satisfactorily continuous along the root of the notch for all but the most sharply notched specimens. For specimens with a notch root radius of 0.001 in. or less, a cadmium plate was not obtained to the base of the notch.

Sustained-Load Tests:

Buttonhead specimens, Figs. 1(*a*) and 1(*b*), were loaded on creep rupture machines to determine the response to sustained-load applications of the respective steels. Suitable aligning fixtures were employed to ensure concentricity of load application.

A limited number of 0.9-in. diameter specimens (stress concentration $K = 5$), Fig. 1(*c*), were also tested. These specimens were tested in a suitable aligning fixture in a 300,000-lb capacity testing machine. The load was maintained by manual adjustment of the machine, while tests were limited to a maximum duration of one day.

Hydrogen Analyses:

Suitable sections of electroplated specimens were submitted to the National Research Corp., Cambridge, Mass., for hydrogen determination. The hydrogen-bearing specimens, immediately after electroplating, were cooled in dry ice and were shipped by air freight. Specimens were kept at the reduced temperature until analyzed. The treatment employed is one which has been used to prevent diffusion of the hydrogen (10) into the specimen, and that it is probably effective may be gathered from the fracture results reported by Troiano *et al* (11).

Metallography:

All fractures were regularly examined visually and at low magnifications. Specimens that remained unbroken after testing were, in selected instances,

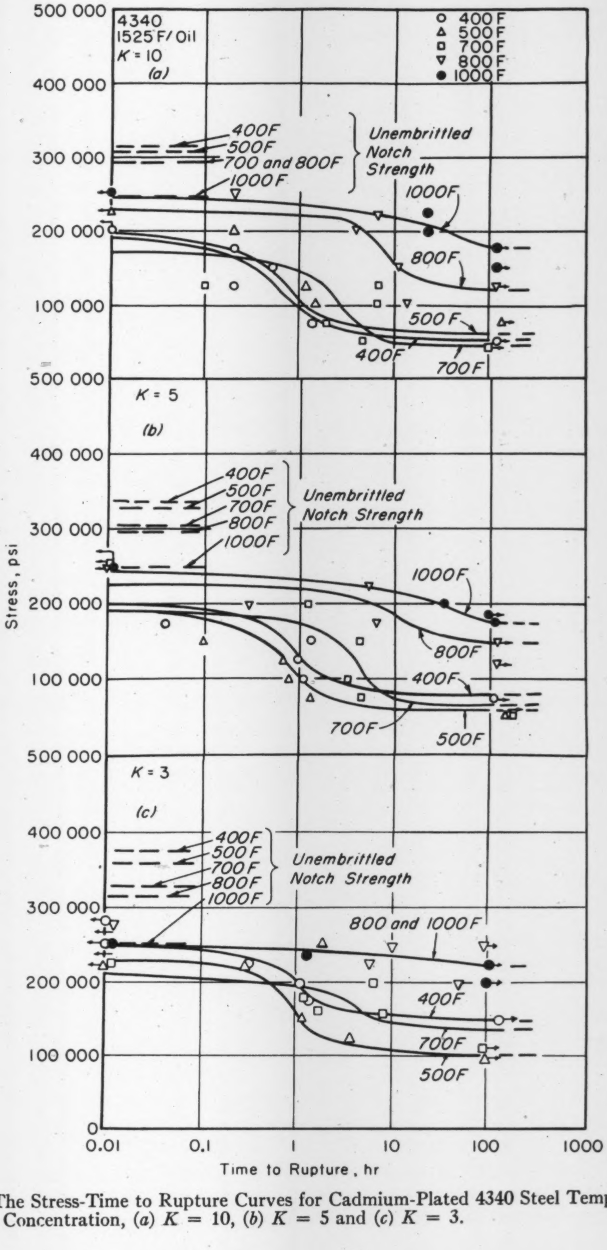

FIG. 2.—The Stress-Time to Rupture Curves for Cadmium-Plated 4340 Steel Tempered as Indicated, Stress Concentration, (*a*) $K = 10$, (*b*) $K = 5$ and (*c*) $K = 3$.

sectioned longitudinally and examined for fracture development at the base of the notch.

Representation of the Test Data:

In the development of the text of the report, reference is made to characteristic curves and summary plots of the data. Additional data are presented by means of photographs.

developed by Petch and Stables, the notch in a sharply notched specimen constitutes the Griffith crack. As was pointed out earlier, this crack has been observed experimentally to develop and grow. However, as the notch root radius is increased, it may be expected that the notch eventually will no longer function as a Griffith crack. For such experimental conditions the fracture process due to the action of hydrogen may be expected to be different from that described for the very sharp notch. In addition to this, relatively mild notch conditions characterize service applications. Sustained-load tests, therefore, are needed to establish the effect of notch sharpness on hydrogen induced crack development.

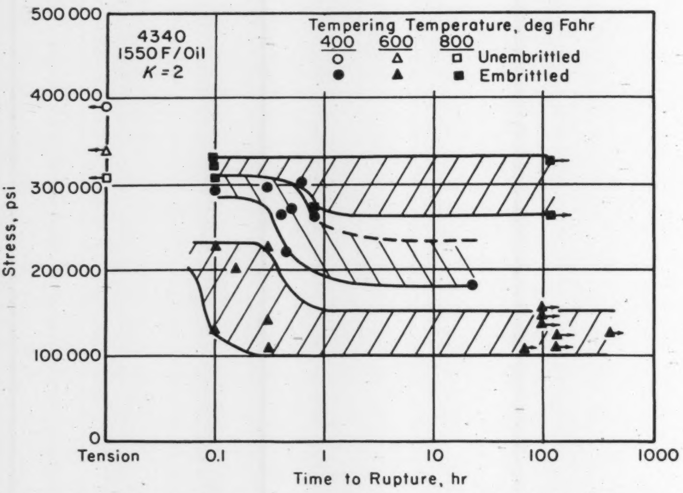

FIG. 3.—The Stress-Time to Rupture Curves for Cadmium-Plated 4340 Steel Tempered as Indicated, Stress Concentration, $K = 2$.

EXPERIMENTAL RESULTS

Hydrogen Analyses of the Steels Studied:

Hydrogen analyses completed on cadmium-plated sustained-load specimens indicated that:

1. The average hydrogen content was ~2.5 ppm; and

2. Within the scatter of the data, steel composition had no effect on the amount of hydrogen contained in the specimen after cadmium-electroplating.

Sustained-Load Tests:

According to the Griffith-Orowan theory of sustained-load failures as

In all, seven steels have been studied. However, it is necessary to discuss in detail the results obtained for only one steel, as the variables introduced affect the different steels in much the same way. 4340 steel has been selected for detailed examination.

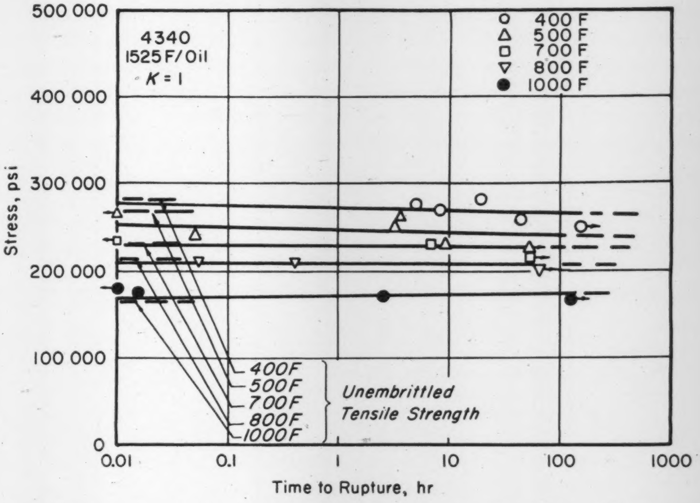

FIG. 4.—The Stress-Time to Rupture Curves for Cadmium-Plated 4340 Steel Tempered as Indicated, Stress Concentration, $K = 1$.

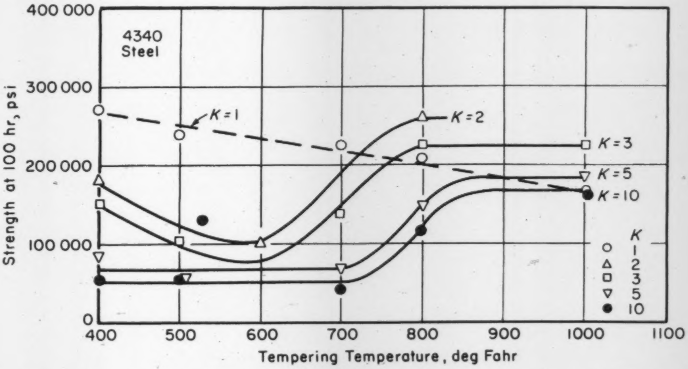

FIG. 5.—The Rupture Strength at 100 hr *versus* Tempering Temperature with Stress Concentration as Parameter for Cadmium-Plated 4340 Steel.

4340 Steel.—Under the most severe notching conditions, 4340 steel was found to be subject to sustained-load failures at all strength levels examined (Fig. 2(*a*)). After tempering at 400, 500, and 700 F, nearly the same load *versus* "time to rupture" curves were obtained and, in general, failure occurred after a loading time of less than 10 hr at a nominal stress of from 50,000 to 75,000 psi.

Tempering at 800 and 1000 F led to a displacement of the stress *versus* "time to failure" curves to the right and to higher stresses. The indicated improvement in properties, therefore, increased progressively as the tempering temperature was raised from 700 F.

For the stress concentration of 10, sustained-load failures due to the action of hydrogen introduced on cadmium-electroplating are very serious for 4340 steel tempered at from 400 to 700 F, inclusive. Reduced properties can be expected from this source after tempering at 800 F. Even after tempering at 1000 F, failures arising from the action of electrodeposited hydrogen may be expected.

For specimens containing a notch leading to a theoretical stress concentration of 5, only slightly modified results are obtained (Fig. 2(*b*)). But as the stress concentration is reduced to 3 (Fig. 2(*c*)) rather marked changes in the series of curves take place.

Perhaps the most significant of these changes is that associated with the minimum fracture stress plotted in Fig. 5. After tempering at from 400 to 700 F, a nearly constant minimum stress to produce failure is measured for stress concentrations of 10 and 5. At higher tempering temperatures, the minimum stress is raised. At a stress concentration of 3, a minimum is indicated in the "minimum strength" *versus* tempering temperature curve (Fig. 5). This occurs at a tempering temperature of about 600 F. At this stress concentration ($K = 3$) increased resistance to hydrogen embrittlement is obtained by tempering at 400 F or at 700 F and above. There is then evidence of the 500-embrittlement phenomenon for the mildly notched specimens.

The trend of the data for a stress concentration of 3 is established with greater emphasis at a stress concentration of 2 (Figs. 3 and 5). The data reported in this last set of curves were obtained for a different heat of 4340 steel (13), but, none the less, are properly ordered in the present comparisons. Finally, for smooth specimens, that is, $K = 1$, while sustained-load failures occur (Fig. 4) such failures take place with little drop in stress. The breaking stress at 100 hr (S_{100})[5] *versus* tempering temperature curve, Fig. 5 thus differs little from that measured for unembrittled smooth specimens.

In Fig. 6 the breaking stress at 100 hr is plotted against stress-concentration with tempering temperature as parameter. Included in the figure are the strength values for nonplated specimens. It is immediately evident that cadmium plating has introduced major changes in the notch properties of the steel studied for all tempering conditions and for all conditions of notching. In this plot the strength of smooth specimens is indicated as unaffected by the presence of hydrogen.

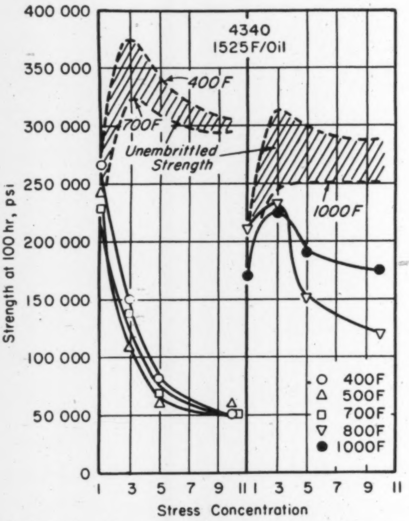

FIG. 6.—The Rupture Strength at 100 hr *versus* Stress Concentration with Tempering Temperature as Parameter for Cadmium-Plated 4340 Steel.

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[5] S_{100} is an extrapolated breaking load for a holding time of 100 hr.

After tempering at 400, 500, and 700 F, the minimum strength (S_{100}) of plated specimens with increasing stress concentration drops abruptly from the values measured for the smooth specimens. This is in contrast to the indicated behavior of the nonplated specimens. Further, little numerical difference is noted for the three tempered conditions.

For specimens tempered at 800 and 1000 F, on the other hand, the minimum strength of plated notched specimens, in keeping with the trends measured for the nonplated specimens, first increases with increase in the stress concentration and then at high values of stress concentration is reduced. In conformance with the concept that the action of hydrogen in the steel is to effectively reduce the cross-section, the trend of these data is that expected.

In brief summary, the above sustained-load data show that a profound difference in fracture behavior may be expected from plating 4340 steel which has been tempered at from 400 to 700 F. For this steel tempered at 800 and 1000 F, the sustained-load data, at first inspection, are understandable in terms of a section reduction arising from the action of hydrogen in the steel. Thus, for the notched specimens, the qualitative trends of the data are similar to those for the nonplated specimens.

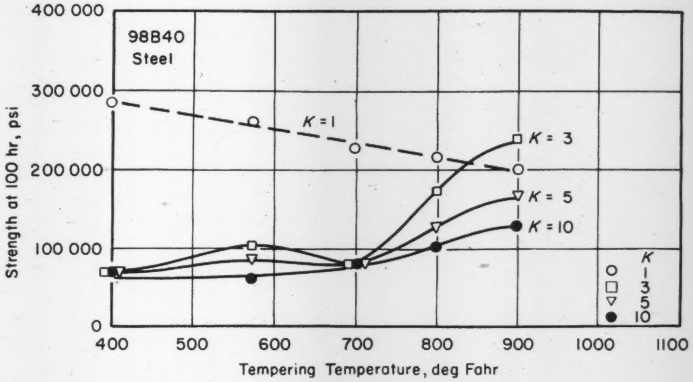


FIG. 7.—The Rupture Strength at 100 hr *versus* Tempering Temperature with Stress Concentration as Parameter for Cadmium-Plated 98B40 Steel.

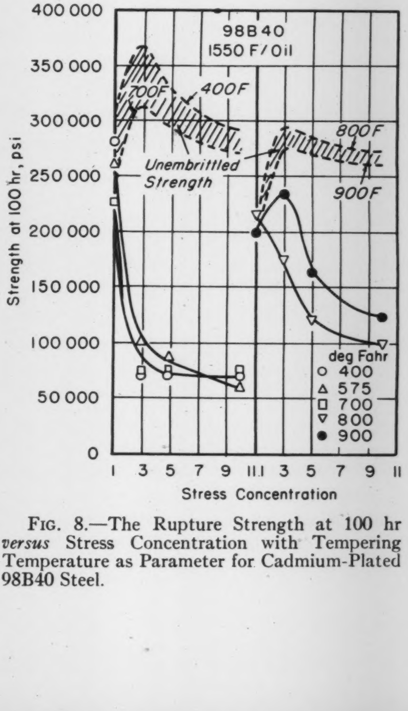


FIG. 8.—The Rupture Strength at 100 hr *versus* Stress Concentration with Tempering Temperature as Parameter for Cadmium-Plated 98B40 Steel.

The quantitative differences would then be due to the reduction of the load-bearing section of the specimen through hydrogen induced crack development. The questions of crack development in smooth specimens and the fracture behavior of specimens tempered at and below 700 F are discussed in a later section.

98B40 Steel.—The notch strength (S_{100}) of plated specimens as a function of the tempering temperature is presented in Fig. 7 with the stress concentration as parameter. These data with certain variations are in agreement with the results obtained for 4340 steel. Of principal interest are the facts that the 98B40 steel studied is more sensitive to the action of hydrogen in leading to embrittlement for a tempering temperature of 400 F, and that in contrast to 4340 steel the tempering temperature range characterizing greatest sensitivity to hydrogen extends to 800 F and possibly higher. These trends are best shown in Fig. 8. Thus at tempering temperatures of 800 F and below, the notch strength of plated specimens drops rapidly with increasing stress concentration from the strength measured for the smooth specimens. Tempering at 900 F promotes an initial increase in the notch strength with increase in stress concentration followed by the expected notch strength reduction at the highest stress concentrations.

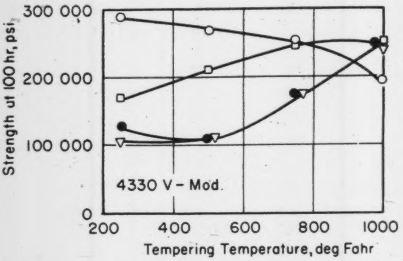


FIG. 9.—The Rupture Strength at 100 hr *versus* Tempering Temperature with Stress Concentration as Parameter for Cadmium-Plated 4330 Vanadium-Modified Steel.

4330 Vanadium-Modified Steel.—The notch strength (S_{100}) of plated specimens as a function of the tempering temperature is presented in Fig. 9 with the stress concentration as parameter. The notch strength (S_{100}) as a function of stress concentration with the tempering temperature as parameter is presented in Fig. 10. These data clearly are related to the data obtained for both the 4340 and 98B40 steels. Thus by the discussion earlier developed in considering the data for 4340 steel, the 4330 Vanadium-Modified steel becomes susceptible to hydrogen embrittlement at a tempering temperature of 750 F and becomes increasingly embrittled as the tempering temperature is reduced. While the tendency to develop the embrittled condition exists at all but the highest tempering temperatures, the degree of embrittlement for this steel is less than

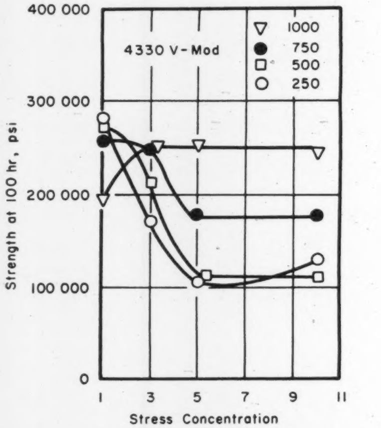


FIG. 10.—The Rupture Strength at 100 hr *versus* Stress Concentration with Tempering Temperature as Parameter for Cadmium-Plated 4330 Vanadium-Modified Steel.

that observed for both the 4340 and 98B-40 steels.

The behavior in the sustained-load test of the three steels 4340, 98B40, and 4330 Vanadium-Modified suggests that for a series of steels of related composition and tempered to a comparable tensile strength, the order of increasing susceptibility to hydrogen embrittlement is the order of increasing carbon content.

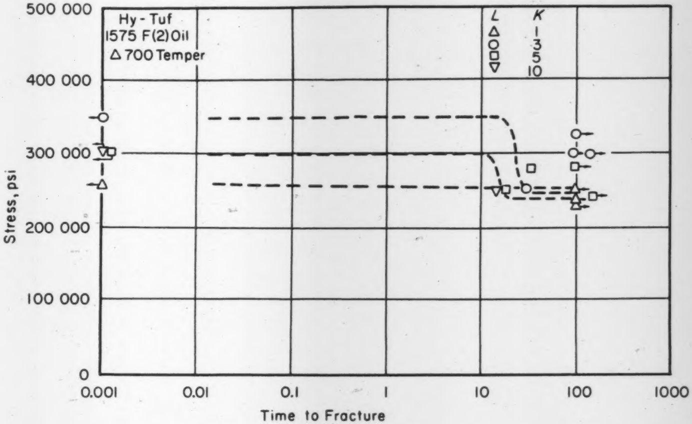


FIG. 11.—The Stress - Time to Rupture Curves at Indicated Stress Concentration for Cadmium Plated Hy-Tuf Steel Tempered at 700 F.

Hy-Tuf Steel.—The three steels 4330 Vanadium-Modified, 4340, and 98B40 are low-silicon bearing steels forming a characteristic group. Three steels alloyed with about 1.5 per cent silicon form a group with characteristic properties that are qualitatively different from those measured for the first three steels. Thus the 1.5 per cent silicon materials are characteristically relatively insensitive to pronounced embrittlement through the action of electrodeposited hydrogen, as is emphasized in the creep-rupture data for Hy-Tuf steel presented in Figs. 11 and 12. This steel at a strength level of 230,000 psi, while subject to sustained-load failures, suffers such failures only when the applied loads are high and sustained for a long period of time. This steel, therefore, is only slightly embrittled through cadmium-electroplating.

Crucible UHS-260 Steel.—The 1.5 per cent silicon steels as a group develop tensile properties that are modified only slightly for relatively wide changes in tempering temperature. The UHS-260 steel available for the present study was heat treatable to strength levels of 250,000 psi and 270,000 psi. This steel heat treated to these strength levels provides much needed data to bridge the large gap between the tensile strengths measured for Hy-Tuf and Tricent steels. The notch strength (S_{100}) *versus* tempering temperature and the notch strength *versus* stress concentration data are presented in Figs. 13 and 14.

These data indicate that UHS-260 like Hy-Tuf is relatively insensitive to the embrittling action of electrodeposited hydrogen. It is interesting to observe that for this steel susceptibility to em-

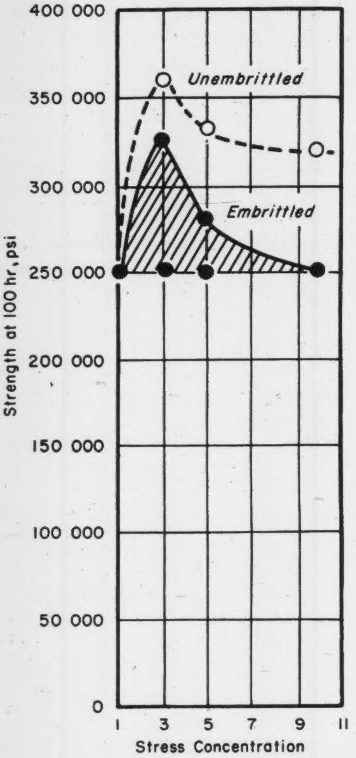


FIG. 12.—The Rupture Strength at 100 hr *versus* Stress Concentration for Cadmium-Plated Hy-Tuf Steel Tempered at 700 F.

brittlement increases as the tempering temperature is raised from 550 to 800 F.

Tricent Steel.—The notch strength (S_{100}) of plated specimens as a function of the tempering temperature is presented in Fig. 15 with the stress concentration as parameter. The response of this steel to the action of hydrogen is complex and at the present time no reasons for this behavior can be advanced. Thus for mild notch conditions ($K = 3$), maximum resistance to hydrogen induced failures obtains after tempering at 400 F. For the intermediate notch severity ($K = 5$) optimum properties are obtained after tempering at 550 F. For the sharpest notch, properties at all tempering temperatures are equal. The notch strength (S_{100}) rapidly drops from the stress measured for the smooth specimen as the notch severity is increased, Fig. 16.

Super TM-2 Steel.—This steel was studied at a strength level of 275,000 psi obtained by tempering at 500 F. The notch strength (S_{100}) data are presented in Fig. 17.

Sustained-Load Strength versus Tensile Strength:

The trends of the data for the several steels may be profitably appraised

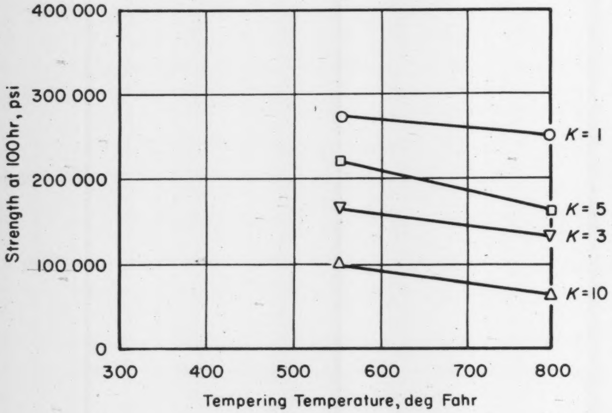


FIG. 13.—The Rupture Strength at 100 hr *versus* Tempering Temperature with Stress Concentration as Parameter for Cadmium-Plated Super Hy-Tuf Steel.

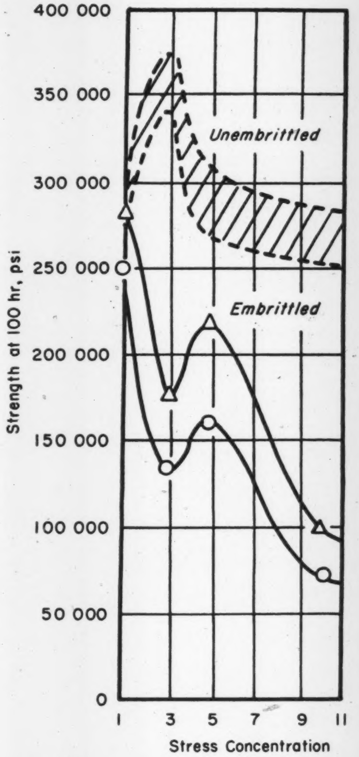


FIG. 14.—The Rupture Strength at 100 hr *versus* Stress Concentration with Tempering Temperature as Parameter for Cadmium-Plated Super Hy-Tuf Steel.

when plotted against tensile strength while the stress concentration factor is held constant as in Figs. 18(*a*)(*b*)(*c*) to 22. These data have an immediate application in the evaluation of the properties of small sections of the steels heat treated to the pertinent strength levels.

Sustained-Load Strength versus Specimen Size:

As has been pointed out, the effects of hydrogen introduced into the metal as a consequence of electroplating in leading to sustained-load failures should be minimized as the specimen size increases. To examine this prediction a series of sustained-load tests were completed on cadmium-plated 0.9 in. diameter specimens. The limited test results obtained are presented in Fig. 19. Of particular interest is the fact that specimens tempered at 400 F are much more susceptible to the action of electrodeposited hydrogen than are the specimens tempered at 600 F. These results are compared in Fig. 20 with data obtained for the plated 0.3-in. diameter specimens and with data obtained for variously sized nonplated specimens.

In this figure, the pronounced roles of size for the nonplated specimens and hydrogen induced brittleness for the

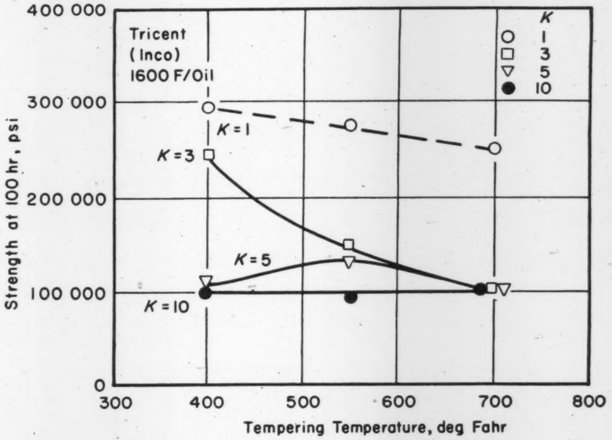


FIG. 15.—The Rupture Strength at 100 hr *versus* Tempering Temperature with Stress Concentration as Parameter for Cadmium-Plated Tricent Steel.

plated specimens are clearly set forth. Since the notch strength values plotted are nominal, the data in this figure are singularly significant.

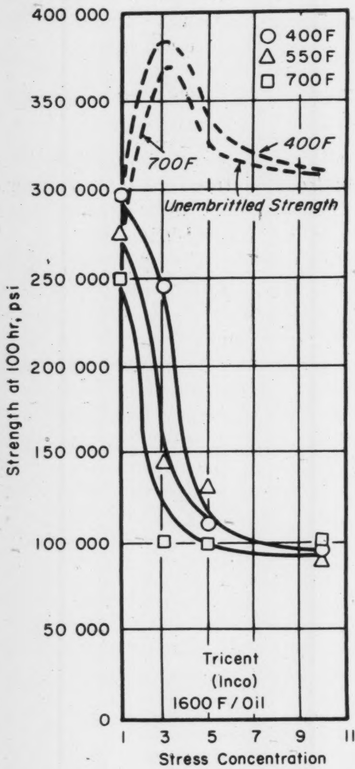


FIG. 16.—The Rupture Strength at 100 hr *versus* Stress Concentration with Tempering Temperature as Parameter for Cadmium-Plated Tricent Steel.

Thus, for the unplated specimens a monotonically decreasing notch strength is measured as the specimen size increases. For the plated specimens, a monotonically increasing notch strength is measured. These two quantities must eventually bound a notch strength which is a limiting quantity for the steel and heat treatment. A transition from a high to a low numerical value is indicated for this quantity, but the data available are not sufficient to allow a complete discussion of this transition at this time.

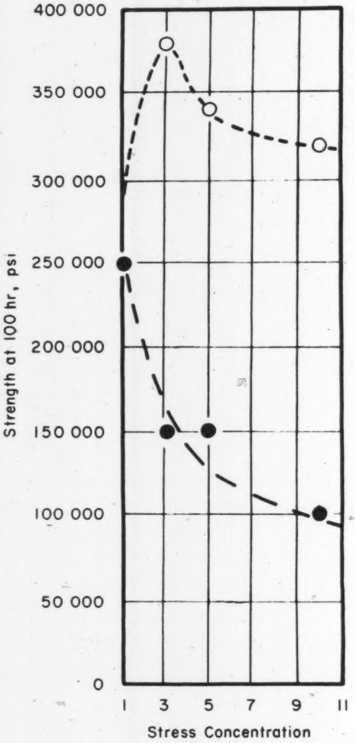


FIG. 17.—The Rupture Strength at 100 hr *versus* Stress Concentration for Cadmium-Plated Super TM-2 Steel Tempered at 500 F.

The sustained-load tests which have been discussed for the small specimens are directly applicable to the evaluation of properties to be expected in service applications of the steels studied when the service members are small. However, it is evident that as the structural member increases in size the adverse effect of hydrogen embrittlement will be minimized. This does not mean, however, that properties of small size unembrittled

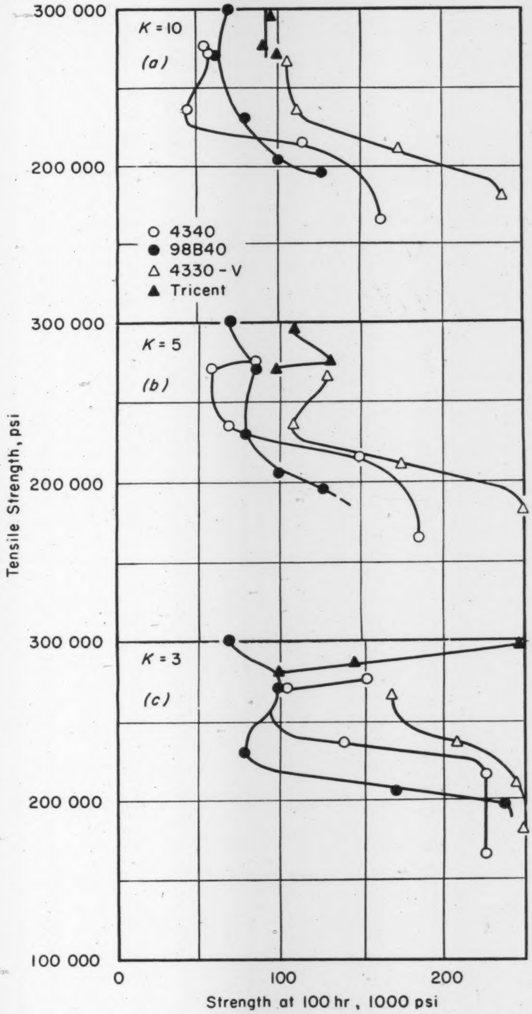


FIG. 18.—The Rupture Strength at 100 hr *versus* Tensile Strength for the Indicated Steels with Stress Concentration, (*a*) $K = 10$, (*b*) $K = 5$, and (*c*) $K = 3$.

specimens will be realized because with increase in section size an adverse section size effect may be introduced. Since the changes in section size affect the notch properties differently for the different steels, it is not possible to propose without reservation the properties to be expected in large electroplated sections. Maximum notch strengths, however, can be estimated from the data obtained in the section size study and are reported in Wright Air Development Center Report TR 56-395 Part 1 (8). Selected data are presented in Fig. 21. These data are offered as probably qualitatively indicating the trends of the notch properties in high-strength electroplated steels. These predicted trends, however, should be checked by suitable tests.

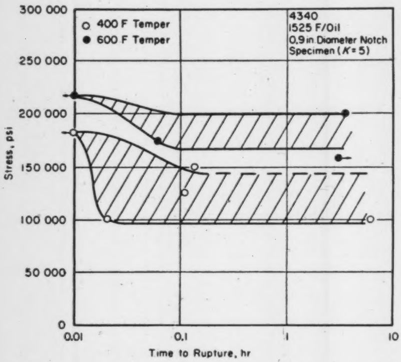


FIG. 19.—The Stress-Time to Rupture Curves for 0.9 in. Diameter Specimens of Cadmium-Plated 4340 Steel, Stress Concentration, K = 5.

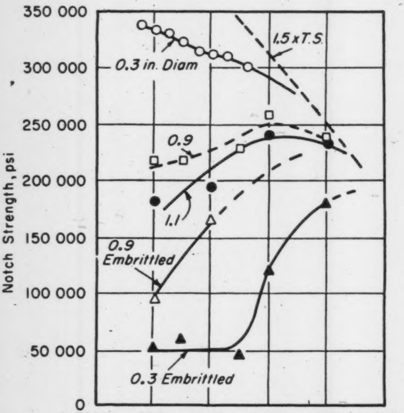


FIG. 20.—The Notch Strength Measured for Embrittled and Unembrittled Specimens of the Indicated Sizes *versus* Tempering Temperature (The Notch Strength for the Embrittled Specimens was Measured under Sustained Loading).

Fracture Development Under the Joint Action of Hydrogen and Sustained Load:

Crack development resulting from the joint action of hydrogen and stress as it has been described previously (3, 4, 5) is essentially from the base of the notch into the core of the specimen. The machined notch, therefore, functions as a Griffith crack and the experimental conditions, that is, hydrogen induced crack growth, lead to the extension of this crack with eventual failure of the section. The conditions here described are not obtained for a smooth or mildly notched specimen, and the failure of these latter type specimens is a point of interest. Two examples of the fractures obtained for smooth-plated specimens are presented in Figs. 22(*a*) and (*b*).

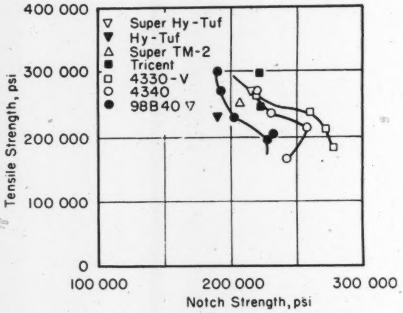
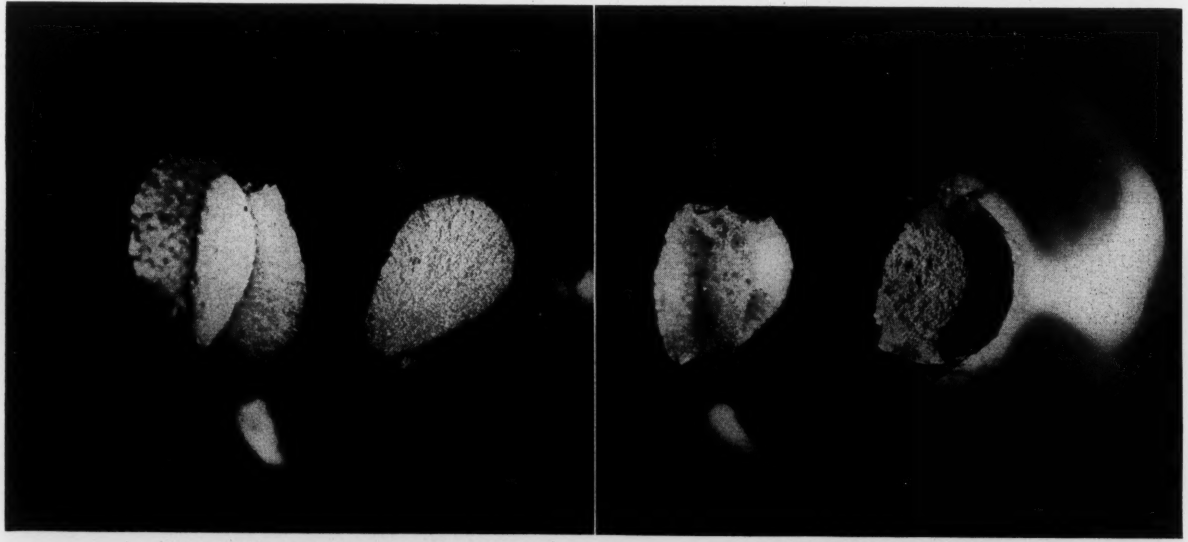


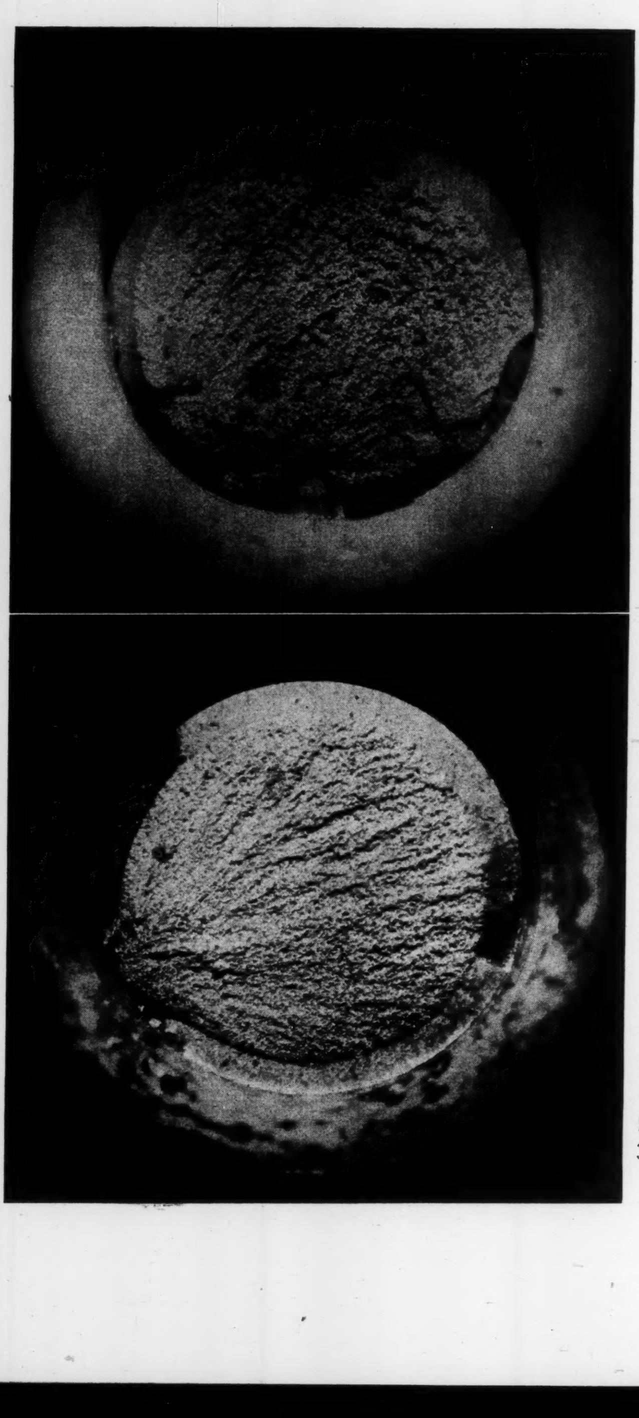
FIG. 21.—The Notch Strength for 0.9 in. Diameter Specimens, K = 10, *versus* Tensile Strength for the Indicated Steels.

While these fractures resulted from the action of hydrogen, at least in point of initiation, it is striking that the preliminary stage of crack development has led to the extension of internal cracks. For Fig. 22(*a*) the crack is clearly nucleated at or near the surface, but for Fig. 22(*b*), there is no obvious connection between the internal crack and the surface until after the fracture



(*a*) Nucleation at or near surface. (*b*) Nucleation subsurface.

FIG. 22.—Sustained-Load Fracture for Cadmium-Plated Smooth Specimen, 4340 Steel.



(*a*) Nucleation at or near surface; tempered at 400 F for 4 hr.

(*b*) Nucleation subsurface; tempered at 600 F for 4 hr.

FIG. 23.—Sustained-Load Fracture for Cadmium-Plated Notched Specimen, $K = 2$.

is completed. Thus for both specimens fracture has taken place due to the separation of zones in the specimen which are expected to be relatively hydrogen free (5). Further, while the broken sections are modified cup-cone fractures, in contrast to what would be expected, the reduction of area (Fig. 22(*a*)) is either nil or very small. This signifies that the indicated shear failures were generated in the hydrogen rich zones with greater ease than could be the failure associated with the maximum normal stress.

The fracture sequence is then describable in stages as follows:

1. *Figure 22*(*a*).—(*a*) A fracture nucleus obtains at or very near the specimen surface;

(*b*) Due to the action of hydrogen this nucleus is activated and grows;

(*c*) The crack surface that develops functions as an effective stress raiser and strengthens the critical section in which it is developed. This strengthening effect is sufficient to arrest further crack development in the absence of hydrogen;

(*d*) Due to the presence of hydrogen, however, the crack continues to develop until it leaves the hydrogen-containing zone, due to mechanical overloading;

(*e*) Further crack development takes place until mechanical equilibrium is established. The crack then stops, and at this stage the internal crack has developed to the degree indicated by the granular fracture surface in Fig. 22(*a*);

(*f*) Final failure takes place when hydrogen has diffused through the remaining intact metal to a sufficient degree to allow precipitation along the indicated shear planes of failure. This hydrogen precipitation is proposed to allow the shear failure to progress without requiring general plastic deformation.

2. *Figure 22*(*b*).—(*a*) For this specimen no surface nucleation of fracture is observed, while the fracture is a cup-cone fracture;

(*b*) Fracturing has taken place after significant plastic strain, as measured by reduction of area;

(*c*) The fracture is considered as differing but little from a normal tension fracture;

(*d*) It is proposed that fracture for this specimen resulted from an overloading of the specimen due to the weakening of the outer fibers of the specimen through the presence of hydrogen.

When specimens containing a mild notch are tested after electroplating, behavior similar to that described for Fig. 22(*a*) may be observed (Fig. 23(*a*)). For this specimen a crack was nucleated under but close to the surface. The crack then developed by internal extension with eventual fracture resulting from the consequent eccentricity of loading with the development of a nearly continuous shear lip about the base of the notch.

That the fracture nucleus may develop under the surface of the notch is emphasized by consideration of Fig. 23(*b*). The small independent fracture nuclei here present clearly do not extend to the surface, but, in the aggregate, have eventually promoted failure.

From the above discussion it may be seen that the fractures resulting from crack formation due to the action of hydrogen arise from the interaction of a complexity of factors. Among these factors are: (*a*) the notch geometry, (*b*) the hydrogen distribution, and (*c*) the role of normal and shear stresses in leading to crack formation.

It is evident from the discussion of Figs. 23(*a*) and (*b*) that mild notches do not function as suitable Griffith cracks. The fracture nuclei which origi-

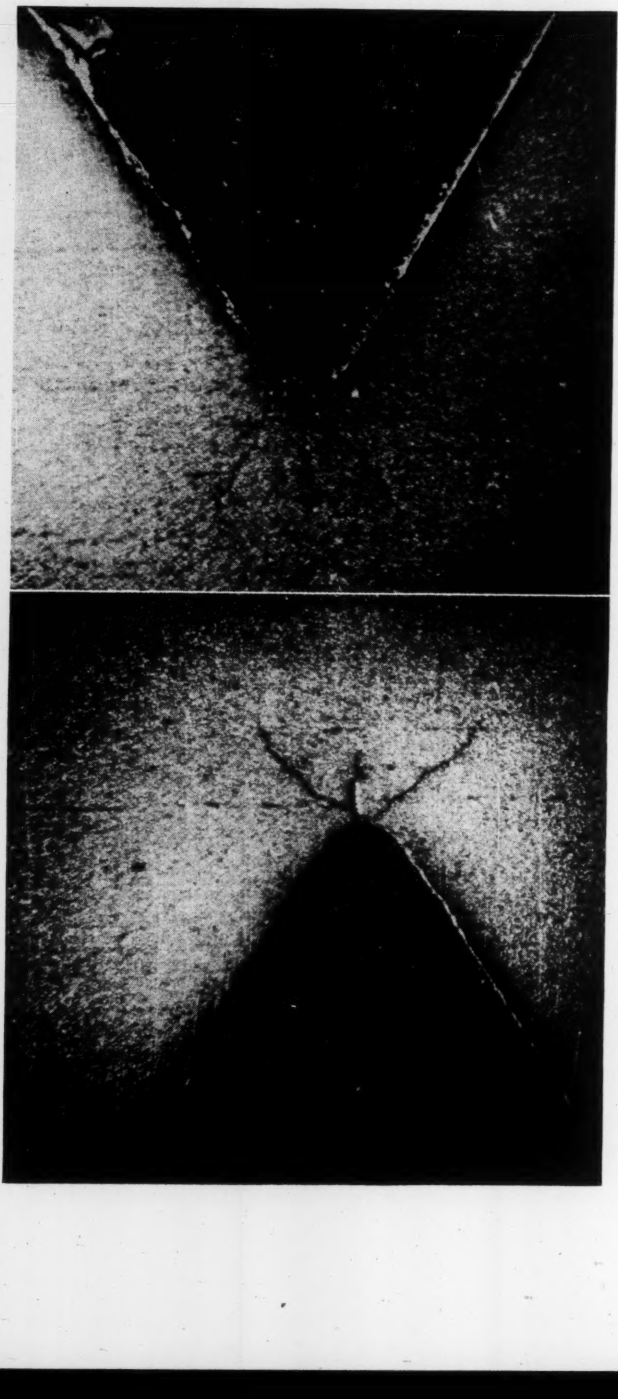


FIG. 24.—Crack Development in Cadmium-Plated 0.9 in. Diameter Specimen, $K = 5$, 4340 Steel Tempered at 600 F.

nate at the surface in smooth and mildly notched specimens probably are associated with tool marks or inclusions. Internal fracture nuclei may be associated with inclusions initially, and their growth can be likened to the development of "fish eyes" associated with hydrogen induced failures in softer steels. However, still another potentially pertinent factor is the condition of triaxiality of stresses.

If a limiting condition of triaxial stress is required to produce fracture, this condition could be established by a suitable inclusion. It potentially could be established also at the cupic point[6] for the stress system induced by the notch. For sharp notches this point would lie close to the surface near the root of the notch. For mild notches this point would be increasingly toward the center of the specimen. Considering the depth of hydrogen penetration immediately after plating (5), for sharp notches the condition of maximum triaxiality would obtain in a hydrogen enriched zone, while for mild notches hydrogen diffusion would be required to reach this point. For a very sharp notch it would be difficult to differentiate between a crack arising through extension of the notch base and one which arises discontinuously by separation at the cupic point and subsequent extension to the surface.

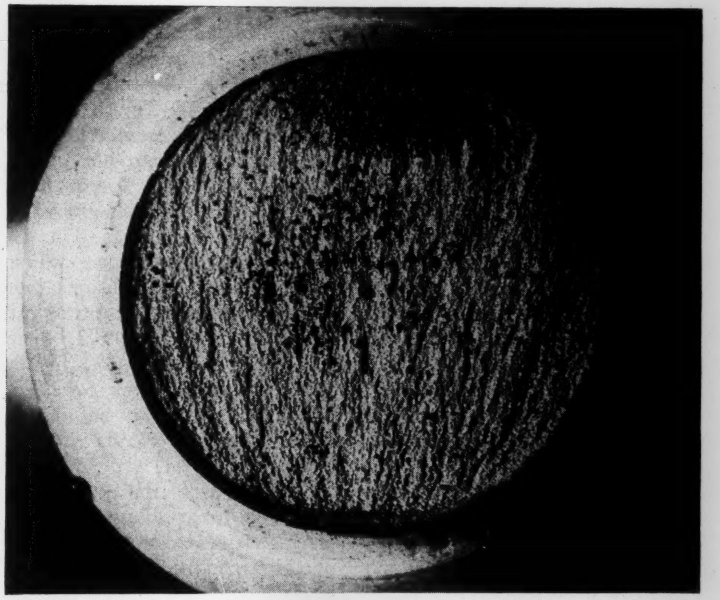


FIG. 25.—Sustained-Load Fracture in 0.9 in. Diameter Cadmium-Plated Specimen (Note Fracture Nucleus and Shear Lip).

Examination of numerous specimens for shear lip development, which may be taken as evidence of subsurface fracture nuclei formation, revealed that a shear lip is frequently obtained even for the most sharply notched specimens. From these data it follows that a hydrogen-induced shear failure regularly accompanies crack formation. Such shear failures can be of two types, the first of which has already been described while the second is indicated in Fig. 24, which is a vertical section through the notch base of a 0.9-in. diameter specimen which had been loaded after cadmium plating.

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[6] In reality a subsurface ring for the type specimens used in this investigation. For a discussion of cupic points see M. M. Frocht, "Photoelasticity," Vol. 1, John Wiley and Sons, Inc., New York, N. Y. (1941).

The stress concentration of 5 was obtained for this specimen with a notch root radius of 0.005 in. For this geometry, the cupic point lies $\sim r$ in. under the notch base, while a condition of maximum shear stress exists at the base of the notch and at ±45 deg into the specimen. Hydrogen fractures are well developed both in the vicinity of the cupic point due to the action of maximum normal stresses at this point and along the lines of maximum shear stresses. These cracks are believed to be of subsurface origin.

For a specimen that generates multiple cracks such as are presented in Fig. 24, it is probable that the notch strength modification due to the action of hydrogen will be relatively small. However, crack development can take place in a second way which is much more serious. Such crack development is indicated in Fig. 25.

For the specimen in Fig. 25, fracture growth progressed at a maximum rate in the vicinity of the nucleus, which apparently was subsurface. The growth of the crack progressed with the development of the nearly semicircular "mirror surface." A consequence of this was the development of progressive eccentricity of loading with eventual cataclysmic crack development from the top region in the photograph to the bottom. This type of loading pattern, crack development, and fracture propagation is probably the most common type leading to structural failures.

SUMMARY

A series of low-alloy steels heat treated to tensile strengths from about 180,000 to 300,000 psi has been studied for susceptibility under sustained loads to embrittlement due to cadmium electroplating. Selected specimens after cadmium electroplating were analyzed for hydrogen content. The results and conclusions are summarized below:

1. Hydrogen introduced into cadmium electroplated specimens may be as high as that introduced through severe cathodic impregnation. The average amount of hydrogen, which varies with specimen dimensions, measured for cylindrical creep-rupture cadmium-plated specimens was ~ 2.5 ppm.

2. By a suitable adjustment of the applied load, all steels for all strength levels examined can be made to fail under sustained loads by cadmium plating.

3. The susceptibility of a steel to sustained load failure after cadmium plating depends on the severity of the notching conditions and on the metallurgical structure of the steel. The steels studied fall into two groups, one of which is comprised of steels which become increasingly insensitive to the embrittling action of cadmium plating with increasing tempering temperature, while the second is made up of steels which possess maximum insensitivity at intermediate tempering temperatures. The first group of steels is silicon dilute; the second is silicon rich.

4. While all steels studied can be made to fail in sustained load after cadmium plating, steels possessing high resistance to this embrittlement can be obtained to strength levels as high as

270,000 to 280,000 psi. Structural control at these strength levels, however, must be ensured.

5. It can be predicted that with an increase in specimen size, susceptibility to sustained-load failures should decrease. The limited number of tests which have been completed support this prediction.

6. Sustained-load failures result principally from the formation of cracks due to the action of electrodeposited hydrogen. Crack formation, however, is a complex phenomenon, and the experimental conditions may be such that crack nucleation may be a surface or subsurface phenomenon, while crack propagation may take place in hydrogen-containing or hydrogen-free metal.

Acknowledgment:

The present research program was supported under U. S. Air Force Contract No. AF 33(616)-2362, S/A 4(56-445), and was administered under the direction of the Materials Laboratory, Directorate of Research, Wright Air Development Center.

Over-all development of the program was materially aided by the efforts of Messrs. E. Dugger and A. W. Brisbane, Project Engineers, both of Wright Air Development Center, in cooperation with Syracuse University personnel.

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DISCUSSION

Mr. S. L. Hoyt.[1]—At Battelle Memorial Institute we did quite a little work some years ago on cracking and retained austenite. This was traced to retained austenite and its slow transformation which resulted in the release of hydrogen under very high pressures and produced the underbead crack. That happened either in the normal course of welding with the E 6010 cellulosic type of electrode, or by heat treating a piece of steel to retain a certain amount of austenite, and then charging it with hydrogen at room temperature. The latter would repeat the performance of underbead cracking.

Those experiments we thought were rather definitive in bringing out the relationship between retained austenite and delayed cracking. At about that time, Mr. Higgins, an English metallurgist, was in this country, and I discussed this with him. He had performed an experiment which consisted in hardening, quenching, and tempering an alloy steel of the type that has been described here, and then loaded it at a fairly high stress. After some delay, cracking developed. He said that was due, like underbead cracking, to the delayed transformation of austenite and the precipitation of hydrogen from it.

The object of my discussion is to raise that question to see if that has been taken into consideration in this work and, if so, to what extent the authors ascribe their results to retained austenite; or on the other hand, if they have seen to it that their test specimens were free from retained austenite so that it is not relevant to the presentation.

Mr. E. P. Klier (*author*).—We have not determined the amount of retained austenite present in the steels after heat treating. Other investigators have shown, however, that the amount of retained austenite in steels of the types studied increases as the silicon content increases. This would seem to argue against the hypothesis of cracking advanced by Mr. Hoyt.

Mr. J. M. Hodge.[2]—There is a question in my mind, as I look at these results, as to what extent these results reflect the sensitivity to hydrogen embrittlement or a change in basic toughness or resistance to brittle fracture. I know that you find that lowering the carbon, or increasing the silicon, both seem to increase this resistance to hydrogen embrittlement with which you are concerned. These also both increase the basic toughness of the material.

Furthermore, increasing the silicon permits using lower carbon to obtain the same strength level; so that, in general, when you are speaking about higher silicon levels, you are at the same time speaking about lower carbon contents, because at these higher silicon levels lower carbons are used to get the same strength level.

I would like some comment on this

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[1] Metallurgical Consultant, Battelle Memorial Institute, Columbus, Ohio.

[2] Research Consultant, Steel Product Development, U. S. Steel, Applied Research Laboratory, Monroeville, Pa.

question as to whether you are evaluating the basic sensitivity to hydrogen embrittlement or a basic toughness.

MR. KLIER.—It is our belief that the silicon added to certain of the steels does lead to an increased resistance to hydrogen embrittlement. On the other hand, a reduction in carbon content leads to increased notch toughness. At this time we are not able to present a mechanism for hydrogen embrittlement in terms of these two factors for our results. We do have certain data bearing on this question but analysis is not at present in final form.

MR. STUART V. ARNOLD.[3]—I was wondering whether the time dependency of the embrittling action would not argue against transformation of austenite to martensite as an explanation. I believe that such transformation is not time-dependent.

MR. KLIER.—We believe that in a generally accepted sense Mr. Arnold's statement is correct. However, Mr. Hoyt, we believe, has in mind a specific mechanism of hydrogen embrittlement based on martensite formation through the action of stress. This possible mechanism should, therefore, be considered, but as is indicated above, we do not think that it is the mechanism operative in the present instance.

MR. WALTER BECK[4] (*by letter*).—The author states that: "The plate thickness obtained was from 0.25 to 0.5 mils." Does that signify a stray in the thicknesses of the platings measured on the notched specimens or were the specimens plated with cadmium to various thicknesses in a range between 0.25 to 0.5 mils?

With reference to the author's statement: "For specimens with a notch root radius of 1 mil or less, a cadmium plate was not obtained to the base of the notch," according to our experience, which is in good agreement with that of the Naval Research Laboratory, it should be possible to cover a notch root radius of 1 mil with a homogeneous plating of cadmium from the cyanide bath. What was the technique used for the plating of notches?

The possibility should be taken into consideration that hydrogen is also absorbed in the cadmium electroplate. Was the specimen which was subjected to hydrogen extraction stripped prior to starting the analysis, or analyzed with the cadmium plating on it?

With reference to the author's statement that "Hydrogen introduced into cadmium electroplated specimens may be as high as that introduced through severe cathodic impregnation," was the unplated specimen impregnated with hydrogen from a sodium hydroxide solution containing the strong poison, sodium cyanide, or treated cathodically in a pure sodium hydroxide solution?

MR. KLIER.—Mr. Beck has raised a number of questions to which I submit the following comments:

1. This is a statement of the accuracy to which we have reported the measurements. Our plating conditions were held constant within reasonable limits as we understand them, and thickness was checked only periodically.

2. Notched specimens were plated using the same set-up as was employed for the smooth specimens. We do not question that the notch base for the sharpest notches can be plated, we point out only that in the present tests this did not take place.

3. The plating was not removed.

4. We had in mind results measured on specimens prepared for us under Mr. Beck's supervision. We understand that no poison was used.

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[3] Staff Advisor, Watertown Arsenal, Hingham, Mass.

[4] Head, Surface Treatment and Corrosion Branch, Aeronautical Materials Laboratory, Naval Air Material Center, Philadelphia, Pa.

MESSRS. E. P. KLIER, B. B. MUVDI, and GEORGE SACHS (*authors' closure*).—In the discussions of our paper references have been made repeatedly to fundamental questions of vast importance. It has not been possible for us to present our work in such a way that the very general frame of reference required for discussion of these questions in the paper would have been justified. Of especial importance are the questions pertaining to the mechanism of hydrogen embrittlement. It has been proposed by Mr. Hodge that hydrogen embrittlement, as we have studied it, may perhaps better be called a study of notch toughness. It is our belief notch toughness is the property that we must be most concerned about, and we have discussed this matter at considerable length elsewhere. However, the subject of notch toughness in its various ramifications is very complex, and it is not possible to discuss this subject satisfactorily at the present time. On the other hand, it is in order to point out that the quantity "notch toughness" is becoming a regularly "suspect" factor in any fracture problem, regardless of the origin of the fracture.

Much work has been done in establishing the mechanism whereby hydrogen causes fracture initiation. For the most part, it has been assumed that hydrogen precipitates and through pressure build-up causes rupture of the metal structure. We do not believe that this mechanism is operative, rather, we suggest that the hydrogen precipitates in a form which might be likened to a metastable iron-hydrogen phase which has low strength. The embrittlement action of the hydrogen is effected on a micro- as opposed to a macro-scale.

The mechanism whereby hydrogen is introduced into the specimen is not at present satisfactorily known. It would appear that the most important time period for hydrogen introduction is at the start of plating—before the plate becomes continuous. Hydrogen entrapped in the plate would be unimportant in leading to embrittlement. In a notched section, the hydrogen content at the base of the notch determines fracture behavior. At the present time, this hydrogen content can only be assumed to equal that at other locations on the specimen surface. Diffusion rates must be known, and as they exist in stress and strain gradients for different alloy compositions and microstructures.

It is our belief that hydrogen embrittlement can be understood only when the phase relations in the iron-hydrogen system are adequately known; plus how these relations vary with composition, and how hydrogen diffuses under the conditions of hydrogen embrittlement. Our work in these various areas is limited in that we have concentrated our effort at a fundamental level into establishing the structures of iron-hydrogen alloys. Our results indicate that the structure in certain iron alloys with hydrogen, in which the hydrogen has been introduced at elevated temperatures, is not homogeneous. Rather, the hydrogen is distributed in lamellar form in both austenite and ferrite. We have not as yet established the distribution of hydrogen introduced during electroplating.

THE LOW-BLOW TRANSITION TEMPERATURE*, †

By George M. Orner[1] and Carl E. Hartbower[1]

Synopsis

An easily executed variation in the standard technique for testing Charpy impact specimens has been developed which permits a separation of the crack initiation and crack propagation stages of the fracture process. The technique requires "initiation" of a crack by means of a low-energy blow and propagation of the crack by a full-capacity blow of the impact-machine pendulum. The transition temperature so obtained has been demonstrated to be independent of specimen size, notch geometry, and other test variables; thus, it is considered to be a material property, having practical engineering significance. The low-blow transition temperature is believed to be the maximum temperature at which an initiating crack can become self-propagating in a thick plate (where the energy required to produce shear lips is small compared with the elastic energy available for crack propagation).

In structural design, an adequate margin of safety must be provided against brittle (catastrophic) fracture. In general, designers attempt this in one of three ways: (*a*) by removing those features of design or fabrication which are known to give rise to stress concentrations and notches; (*b*) by requiring inspection (visual, magnetic particle, and/or X-ray) to assure that inadvertent defects from which fracture may originate will be eliminated; or (*c*) by specifying a minimum notch toughness at the lowest anticipated service temperature. Of these three approaches, the third appears to be essential for critical applications because no matter how good the design or how thorough the inspection, it is virtually impossible to eliminate all forms of notches and defects in practical fabricated structures.

The British Admiralty Advisory Committee on Structural Steel has pointed out two schools of thought in this connection (**1**).[2] One school holds that materials should be sufficiently notch-tough so that a brittle crack will not *initiate* under service conditions even in the presence of the stress-raising defects which are nearly always present in welded structures. The second school of thought holds that the conditions for initiation are unavoidably present in all practical structures, and that, therefore, the essential requirement is that under service conditions of stress and temperature the material should not *propagate* a crack in a brittle manner, and that safety can be insured only by choosing steel capable of resisting brittle crack propagation at the minimum service temperature.

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* Presented at the Sixty-first Annual Meeting of the Society, June 22–27, 1958.

† The opinions or assertions contained in this paper are those of the authors and are not necessarily to be construed as official or reflecting the views of the Department of the Army.

[1] Mechanical Engineer and Physical Metallurgist, respectively, Watertown Arsenal Laboratories, Watertown, Mass.

[2] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 634.

The British point out that the conditions of temperature and stress required to initiate brittle cracks are considerably more adverse than those required to keep cracks propagating. Therefore, the first school believes that the prevention of fracture initiation under service conditions is the best economic approach to the problem. However, it is the observation of the writers that the maximum temperature at which brittle crack initiation can occur is in fact generally higher than that required for brittle crack propagation. Cleavage crack initiation is essentially a local phenomenon resulting from a pile-up of dislocations within a single grain or from the hydrostatic tension produced by strain concentration at a gross defect, and so on. Such local stress conditions can be much more severe than the over-all conditions prevailing throughout the structure. Moreover, the Charpy specimen itself contradicts the idea that the maximum temperature at which a brittle crack will keep propagating is higher than that associated with the initiation of cleavage. In V-notch Charpy impact testing, constraint and strain rate are usually sufficient to initiate cleavage at temperatures considerably *above* that at which a brittle crack will become self-propagating (propagated by the release of elastic strain energy in the material surrounding the crack). This is evidenced by the fact that in most steels the onset of cleavage (that is, the highest temperature at which crystallinity is observed in the Charpy fracture surfaces) occurs at the temperature at which the energy values start to fall off, whereas self-propagating fracture does not occur until a temperature is reached which corresponds to a relatively low energy level.

Thus, to insure that brittle catastrophic failure will not occur, the structural engineer must specify a material that will resist self-propagating cleavage at the lowest anticipated service temperature.

As an outgrowth of a long-range program to evaluate and improve the Charpy impact test, a technique has been developed which permits the determination of the maximum temperature at which a crack can become self-propagating. The following report provides a summary description of the development of the technique (the low-blow technique). Readers who want more detail or who want to see additional plots of data are referred to the individual references upon which this summary report is based.

THE LOW-BLOW TECHNIQUE

What Is Accomplished?

The crack initiation and crack propagation stages of fracture are separated by the technique.

Using a basic concept of transition temperature, the technique permits one to determine a crack propagation transition temperature that is essentially independent of notch and specimen geometry. Thus, by means of the low-blow technique, standard V-notch and keyhole Charpy specimens indicate the same transition temperature.

Adiabatic temperature rise effects associated with the crack initiation stage of fracture are essentially eliminated in so far as they affect brittle crack propagation. The effects of adiabatic temperature rise, although frequently overlooked, are thought to be highly significant in Charpy testing. They are considered to be responsible in large part for the temperature displacement between the V and keyhole notched Charpy transition curves as obtained by the conventional (single blow) method.

Procedural Considerations:

Concept.—Orowan (2) has suggested that one may differentiate between ductile and brittle fracture by noting the manner in which the initiating crack is propagated; a brittle fracture crack is propagated by continuous release of the elastic stress field surrounding the crack, whereas a ductile fracture crack requires plastic deformation work for its extension. The research summarized in this paper is based on this concept. The work was undertaken with the objective of developing a technique for determining the maximum temperature at which brittle (that is, self-propagating) cracking can occur.

Slow Bend.—The application of Orowan's concept to slow-bend testing was accomplished by noting the highest temperature resulting in sudden and complete fracture at maximum load, that is, zero energy after maximum load. The choice of maximum load as a focal point was based on an experimental observation by Raring (3), namely, that the start of crack propagation in the thickness direction of a V-notch Charpy slow-bend bar coincides with the maximum in load as determined from the load deflection diagram. Thus, based on the concept that ductile fracture requires plastic deformation work to propagate the initiating crack, specimens absorbing zero energy after maximum load were considered brittle. The highest temperature permitting such fracture was taken as the transition temperature (4).

It should be noted, however, that when complete brittle fracture occurs at the onset of cleavage, the transition temperature so determined has little significance. This is true for both slow bend and impact tests. For example, in the case of high purity low carbon iron, the transition from high to low energy values is precipitous, with brittle fracture occurring at the onset of crystallinity in the fracture surface. Thus, it appears that constraint and strain rate in the standard V-notch Charpy specimen are not sufficient to initiate cleavage in such a steel until the testing temperature is lowered to a value below that at which cleavage propagates catastrophically. In such a case, the transition temperature is associated with initiation rather than propagation of cleavage. Because initiation of cleavage is highly dependent upon specimen geometry and strain rate, such a transition is not considered significant. Cleavage crack propagation, on the other hand, is independent of these variables in that once a crack is propagating as cleavage the strain rate is very high and propagation is unaffected by specimen geometry except in the immediate vicinity of the free surfaces.

In any test intended for determining a brittle-propagation transition temperature, the initiation of cleavage (crystallinity in the fracture surface) must occur at a temperature above the crack propagation transition in order to insure that the transition observed is not associated with initiation. Once a cleavage crack is initiated in a given test specimen, the strain rate and stress state associated with the dynamic front of the natural crack overshadows the effect of the machined notch, the specimen geometry, the rate of loading and, in fact, all of the test variables involved in the particular test except temperature. Because the initiation of cleavage is facilitated by high rates of loading, the slow bend testing was abandoned in favor of impact to assure that cleavage cracking would be initiated in structural steels above the temperature at which propagation occurs under the action of the elastic strain energy.

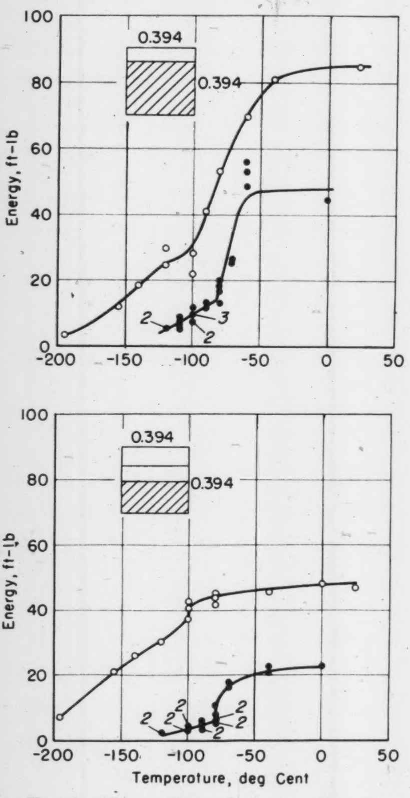

FIG. 1.—Energy-Temperature Transition Curves for Standard Keyhole and V-Notch Charpy Specimens in a Quenched-and-Tempered Class 90 Steel.

The open circles (○) represent energy-to-fracture with a single full-capacity blow; the solid circles (●) represent the energy required to propagate the "low-blow-initiated" crack. Note that the low-blow transition temperature (corresponding to the inflection in the latter curves) occurred at −80 C for both notch types.

Impact.—The application of Orowan's concept to impact testing is accomplished by the use of multiple blows against the standard Charpy specimen (**5**). The first blow consists of a low-energy blow of sufficient magnitude to produce a crack at the apex of the machined notch, thus providing the "initiating crack." The specimen is then fractured in the impact machine using the full capacity of the pendulum. In other words, having introduced the initiating crack by means of a low blow, the specimen is tested in the conventional manner. The crack propagation transition curve is obtained by plotting the energy absorbed under the full-capacity crack-propagating blow *versus* temperature.[3] An abrupt inflection in the low-energy range of this curve is taken to signify the onset of self-propagation for reasons discussed in following paragraphs (Fig. 1) (**6**).

The Initiating Crack.—As stated previously, determination of the brittle crack propagation transition in structural steels requires cleavage crack initiation above the highest temperature at which cleavage can propagate freely. It is the function of the low blow to perform the plastic deformation work which necessarily precedes cleavage crack initiation. Thus, at and even somewhat above the low-blow transition temperature, cleavage is initiated in a recooled specimen by the higher velocity full capacity second blow without further appreciable plastic deformation.

It is stressed that cleavage is not initiated by the low blow. Moreover, it is emphasized that the ductile low-blow-initated crack is not analogous to a machined notch of extreme sharpness. Flebeck and Orowan (7) have shown that cleavage cannot be initiated without copious local plastic deformation from even an "atomically" sharp arrested brittle crack. The ductile low-blow-initiated crack is believed to be more effective in initiating cleavage than an arrested cleavage crack because of the triaxial stresses (that is, hydrostatic tension of plastic constraint) generated as a result of the deformation of a comparatively large volume of metal at the tip of the ductile crack. The volume of deformed metal at the tip of an arrested cleavage crack, on the other hand, is small and is therefore less effective in raising the flow stress to the cleavage fracture level.

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[3] In the initial development work, transition behavior was evaluated by plotting the differences between lateral-expansion measurements taken before and after the crack-propagating blow *versus* temperature. The use of lateral expansion was suggested by the simple relationship that exists between it and energy and the obvious connection between lateral expansion and plastic-deformation work. However, it was later found that plots of energy *versus* temperature provided the same transition temperature as deformation *versus* temperature when using the low-blow technique (**6**).

In impact, the extent of the initiating crack is a function of the magnitude of the low blow used. This raises questions as to the effect of small variations in the initiating crack on low-blow transition temperature determinations. In other words, if the "initiating crack" is shallow or extends an appreciable distance through the specimen, will the test results be significantly affected? Tests indicate that a variation in the extent of the initiating crack in a given steel is not critical providing the low blow is sufficient to produce a well-defined ink-stained crack.[4] If the crack is too small, a large amount of plastic deformation work is required in some steels to initiate cleavage cracking when the second full-capacity blow is struck and, therefore, adiabatic temperature rise and other effects may result in a fictitiously low transition temperature. However, when the crack-initiating low blow is increased somewhat over that necessary to produce perceptible ink staining, the transition temperature is raised only slightly, if at all. For example, in the case of one steel, when the magnitude of low blow was varied (8, 12, 15 and 25 ft-lb), the low-blow

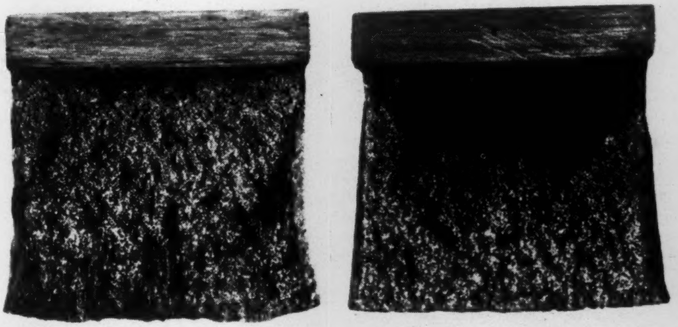

FIG. 2.—Ink Staining Resulting from Impact with 12 and 25 ft-lb Low-Energy Blows on Standard V-Notch Specimens in a 3.3 per cent Nickel Steel.

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[4] India ink containing 10 to 20 per cent by volume of Kodak Photoflo solution as a wetting agent is used as a stain to determine the presence and extent of the initiating crack. Twenty-four hours at room temperature is allowed for the ink to dry.

transition temperature was essentially constant (−90, −80, −80 and −70 C, respectively) (8). The low-energy blows of 12, 15 and 25 ft-lb produced ink staining in this particular steel, whereas, the 8 ft-lb blow did not (Fig. 2).

Temperature of Low Blow.—To avoid possible complications in interpretation, in the early work the crack-initiating low blows were delivered at the same temperatures as those used for crack propagation. Moreover, in the early work, attention was focused on the maximum temperature at which a low blow of crack-initiating energy only produced complete fracture; that is, the crack was propagated by the elastic strain energy developed by the initial blow. It was later recognized that adiabatic temperature rise effects and the low strain rates associated with low-energy blows could, in some instances, delay the initiation of cleavage cracking, and as a result fictitiously low transition temperatures were recorded when attention was focused on the maximum temperature at which complete fracture occurred under the low blow (see Table I). Therefore, the maximum temperature at which complete fracture occurred as a result of the low blow was abandoned as a useful criterion. Because it was now desirable to avoid fracture under the initial low blow, the effect of delivering all crack-initiating low blows at room temperature was investigated. Comparisons between the low-blow transitions obtained with the low blows delivered at the temperature of crack propagation and with low blows delivered at room temperature did not reveal any significant differences between the two methods (9). Thus, a simplification in test procedure was possible; having established at room temperature the approximate low-energy blow required to initiate perceptible ink staining, the complete set of specimens are precracked by the crack-initiating low blow, all at room temperature.

TABLE I.—TRANSITION TEMPERATURES.

Steel	Condition	Rockwell Hardness	Low Blow	Transition Temperature, deg Cent	
				a	*b*
Nickel (3.3 per cent)	Pearlitic	B 82	12	−120	−80
Class B	Pearlitic	B 83	19	0	0
Class 90	Martensitic	B 99	30	−120	−80
Nickel (9 per cent)	Martensitic	C 21	20	..	−160
4340	Martensitic	C 27	30	−120	−130

[a] Based on the highest temperature permitting complete fracture as a result of the initial low blow.

[b] Based on the inflection in the energy to propagate curve.

Criterion of Brittle Behavior.—The early developmental work used lateral expansion as a performance criterion because it provided an indication of the plastic deformation work associated with fracture. Moreover, lateral expansion attending the initial blow provided a measure of the plastic deformation work done in initiating the crack, and the difference between the lateral expansion measurements taken before and after the fracturing blow provided a measure of the work done in propagating the crack.

In the early work, measurements were made with a standard micrometer. Later work, using an improved measuring device which permitted greater accuracy in determining lateral expansion, revealed an abrupt change of slope in the low-temperature region of the crack propagation transition curve (somewhat above the temperature at which complete fracture occurred as a result of the intial low blow). The change of slope indicated a sudden arrest in the rate of decrease of plastic deformation work with decreasing temperature. Plots of energy to propagate *versus* temperature also showed an inflection point at the same temperature at which the inflection occurred in the deformation temperature transition curves. Therefore, a further simplification in the procedure was possible, namely, the use of energy to propagate as read directly from the dial of the impact testing machine to eliminate the need for lateral expansion measurements (6).

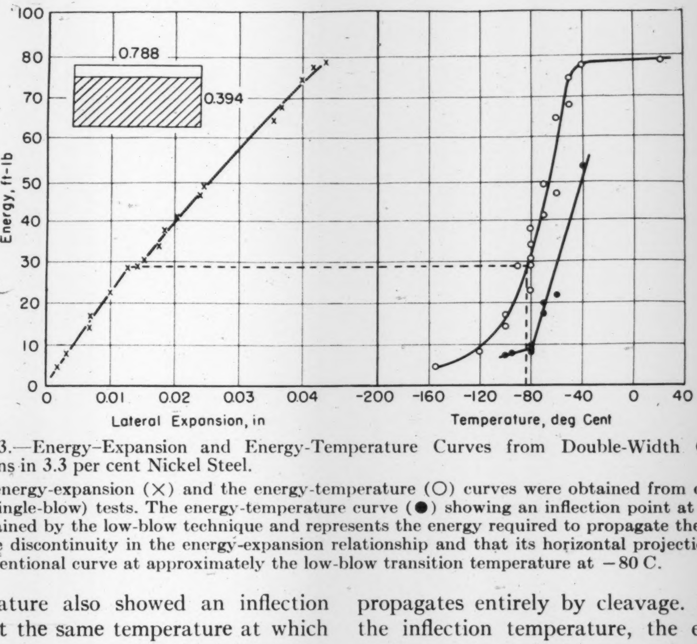

FIG. 3.—Energy–Expansion and Energy-Temperature Curves from Double-Width Charpy Specimens in 3.3 per cent Nickel Steel.

The energy-expansion (×) and the energy-temperature (○) curves were obtained from conventional (single-blow) tests. The energy-temperature curve (●) showing an inflection point at −80 C was obtained by the low-blow technique and represents the energy required to propagate the crack. Note the discontinuity in the energy-expansion relationship and that its horizontal projection cuts the conventional curve at approximately the low-blow transition temperature at −80 C.

Meaning of the Inflection Point.—Based on an examination of crack propagation data and of specimen fracture surfaces, it has been observed that the energy to propagate and the percentage of fibrous fracture decrease rapidly and almost linearly with temperature from the onset of crystalline fracture down to the temperature corresponding to the inflection. At the inflection temperature, the fracture faces exhibit ductility at their outer edges only, where because of the proximity of the free surfaces triaxiality cannot exist; and in the center portion of the specimen the crack propagates entirely by cleavage. Below the inflection temperature, the energy to propagate and the area of ductile fracture continue to decrease proportionately, but at a lower rate. The decrease in energy above the inflection is considered to represent the decrease in work required to propagate the crack in the central portion of the specimen; at the inflection temperature the work to propagate approaches zero. Below the inflection, the energy registered on the Charpy machine is considered to represent elastic energy and the work

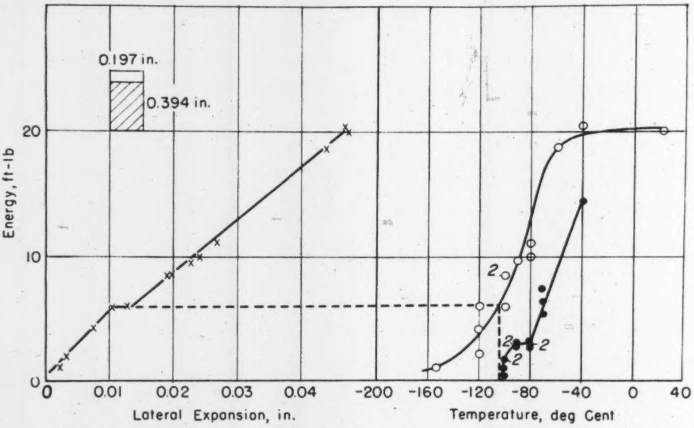

FIG. 4.—Energy-Expansion and Energy–Temperature Curves from Half-Width Charpy Specimens.

Note that the energy level at the discontinuity in the energy-expansion relationship (×) corresponds to a temperature of approximately −100 C on the conventional (single-blow) transition curve (○), and that this temperature is considerably (approximately 20 C) below the temperature corresponding to the inflection in the low-blow transition curve (●).

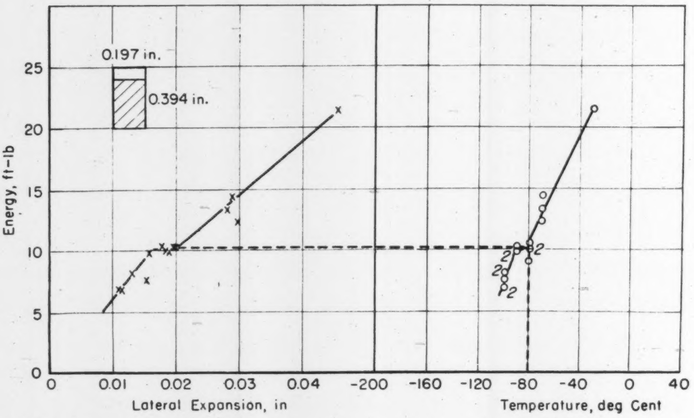

FIG. 5.—Energy-Expansion and Energy-Temperature Curves from Half-Width Specimens Tested by the Low-Blow Technique.

The energy and lateral expansion data represent the sum of the values obtained from the low blow and the crack-propagating blow for each specimen. Note that the energy level at the discontinuity in the energy-expansion relationship (×) corresponds to the low-blow transition temperature of −80 C for this steel. The effects of adiabatic temperature rise from energy absorbed during crack initiation were eliminated from the crack propagation stage of fracture by recooling the specimens before striking the second blow.

required to propagate the crack in the areas where triaxiality cannot exist (at the free surfaces where shear lips occur). The inflection is therefore considered to correspond to the maximum temperature at which elastic energy is sufficient to propagate a cleavage crack in areas where triaxiality can exist (that is, in the center section of the specimen away from the free surfaces, or in a cross-section of infinite dimensions).

Evidence of the validity of the above hypothesis was obtained when it was found that four cross-sections (standard-depth bars with half-width, full-width, and double-width cross-sections as well as half-depth double-width specimens) and three notch types (standard Charpy V and keyhole notches and a half-depth V notch) indicated the same low-blow transition temperature (**6**, **9**). In other words, the inflection point occurred at the same temperature for each of the notch and specimen geometries; thus, the low-blow transition temperature is indicated to be independent of notch and specimen geometry within the limits of notch acuity and specimen size investigated.

Supporting evidence that the Charpy low-blow transition temperature is a material property, independent of variables introduced by the testing technique, was obtained from deformation-energy plots (**10**). The results from conventional (single-blow) Charpy impact tests plotted to show the relationship between energy and lateral expansion revealed an abrupt discontinuity in the low-energy range of the expansion-energy relationship (Fig. 3). The temperature, as read from the conventional energy-temperature curve, corresponding to the energy level at the discontinuity in the expansion-energy curve was observed to correlate quantitatively with the low-blow transition temperature. The discontinuity in the expansion-energy curve is believed to occur at the highest temperature at which cracking becomes self-propagating (see Reference (**10**) for the reasoning upon which this observation is based).

Additional data showing the discontinuity in the energy-expansion curve for a half-width V-notched specimen are given in Figs. 4 and 5. Note from Fig. 4 that the discontinuity in the energy-expansion curve corresponds to a temperature approximately 20 deg lower than the temperature corresponding to the inflection in the energy to propagate curve. This displacement is considered to be due to adiabatic temperature rise occurring as a result of the plastic deformation work done during crack initiation. The fact that such a displacement occurred in the case of the half-width specimen and not with the standard specimen is attributed

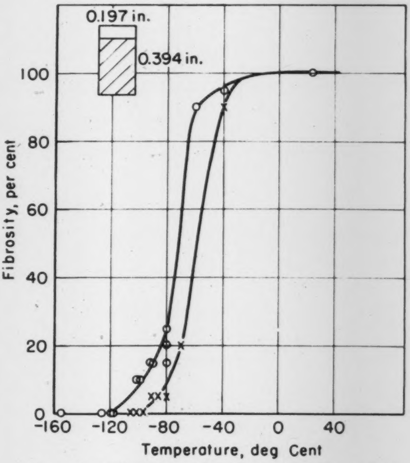

Fig. 6.—Fibrosity–Temperature Transition Curves from Half-Width Specimens Tested by Low-Blow (X) and by Conventional Single-Blow (O) Techniques.

Note that the conventionally obtained transition curve is displaced to lower temperature (approximately 20 C) as a result of adiabatic temperature rise.

to the reduced plastic constraint and, therefore, greater plastic deformation in the half-width specimen. The data for Fig. 5 were obtained from tests on specimens identical to those used for Fig. 4, but tested by the low-blow technique in which the effects of adiabatic temperature rise were eliminated by recooling the specimens before the cracks were propagated. Note that in this case there is excellent agreement between the temperature at the discontinuity in the energy expansion curve and the low-blow transition temperature. Another method of indicating the effect of adiabatic temperature rise is to plot fibrosity temperature curves for specimens tested by both the conventional (single-blow) and the low-blow methods. The effects of adiabatic temperature rise on crack propagation are indicated by the temperature displacement of the two curves. Fibrosity curves for the specimens tested for Figs. 4 and 5 are illustrated in Fig. 6. Note that there is a temperature displacement of approximately 20 deg which correlates with the discrepancy indicated in Fig. 4.

The fibrosity curves in Fig. 7 are presented to show how serious an error can be introduced by neglecting the effects of adiabatic temperature rise. These data pertain to standard keyhole-notch specimens in two steels, namely, a nickel steel requiring 14 ft-lb to initiate a crack and a class 90 steel requiring 28 ft-lb to initiate a crack. Note that at a 10 per cent fibrosity level, displacements of approximately 35 C and 70 C are indicated, and that these displacements are directly proportional to the respective crack-initiating energy values. A question might be raised as to the possibility of strain aging as a factor contributing to the above displacements. In general, strain aging would not be expected in steels

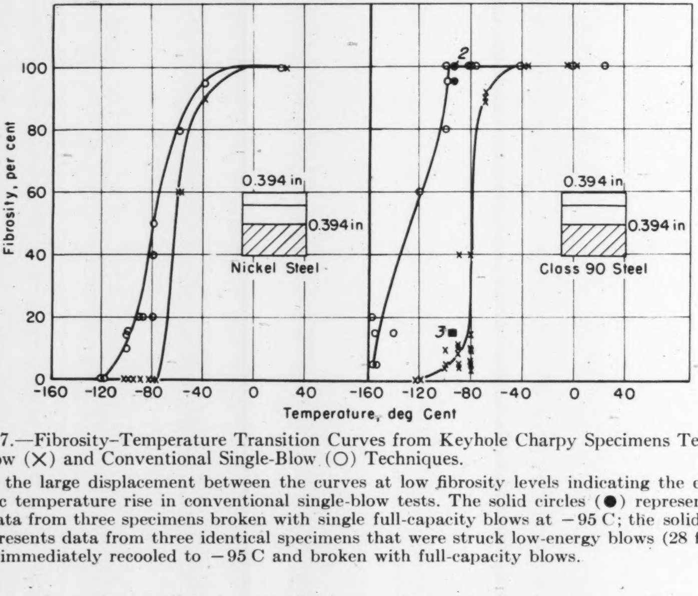

FIG. 7.—Fibrosity–Temperature Transition Curves from Keyhole Charpy Specimens Tested by Low-Blow (×) and Conventional Single-Blow (○) Techniques.

Note the large displacement between the curves at low fibrosity levels indicating the effect of adiabatic temperature rise in conventional single-blow tests. The solid circles (●) represent additional data from three specimens broken with single full-capacity blows at −95 C; the solid square (■) represents data from three identical specimens that were struck low-energy blows (28 ft-lb) at −95 C, immediately recooled to −95 C and broken with full-capacity blows.

of the type investigated, particularly in view of the temperatures involved and short time of storage (approximately 24 hr at 75 F to allow the ink to dry before fracture). To confirm this observation, three class 90 keyhole specimens were tested in such a way as to eliminate the possibility of strain aging; that is, the specimens were delivered the 28 ft-lb low blow at −95 C, immediately reimmersed in the bath at −95 C and then fractured within 15 min. Three additional identical specimens were broken with single full capacity blows also at −95 C. The fibrosity data are plotted for all six specimens in Fig. 7; note that the conventionally impacted specimens broke with practically 100 per cent fibrous fracture compared with only 15 per cent fibrous fracture for the pre-cracked specimens. Thus, strain aging does not appear to have contributed to the curve displacements in Fig. 7.

From an engineering point of view, the low-blow transition temperature is considered to be a basic material property in that it is believed to correspond to the maximum temperature at which the over-all conditions of restraint, strain rate, and so on at the tip of a propagating cleavage crack are just severe enough to maintain cleavage crack propagation in undeformed material.[5]

SUMMARY

The low-blow transition temperature may be determined by an easily executed variation in the standard technique for testing Charpy impact specimens. The advantages of using this technique are:

1. The crack initiation and crack propagation stages of fracture may be separated.

2. Using a fundamental definition of transition temperature, the technique permits one to determine a crack propagation transition temperature that is essentially independent of notch and specimen geometry. Thus, by means of the low-blow technique, standard V-notch and keyhole Charpy specimens indicate the *same* transition temperature.

3. By recooling the specimen before striking the crack-propagating blow, adiabatic temperature rise effects associated with the crack initiation stage of fracture are essentially eliminated in so far as they affect brittle crack propagation. The effects of adiabatic temperature rise, although frequently overlooked, are thought to be highly significant in Charpy testing. They are considered to be responsible in large part for the temperature displacement between the V- and keyhole-notched Charpy transition curves as obtained by the conventional (single-blow) method.

CONCLUSION

The test results summarized above and reported in detail in the various referenced WAL reports indicate that the low-blow transition temperature is independent of test variables and is therefore a material property. The low-blow transition is believed to represent the highest temperature at which self-propagating (catastrophic) cracking can occur in structural steels and is therefore believed to be significant with respect to service casualties.

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[5] It has been demonstrated that a fairly large volume of undeformed material exists in the vicinity of the neutral axis of a deformed Charpy specimen. See Fig. 9, Reference (**6**).

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DISCUSSION

MR. FRANCIS A. BRANDT.[1]—In essence, the authors are proposing that the V-notch Charpy specimen has an insufficiently sharp notch and that this deficiency be overcome by introducing a precrack. I would agree that a sharper notched specimen is required to make the Charpy test meaningful. At the close of the paper the author said, "This is a really significant test." I would wish to hear further ramifications on this statement. He has arrived at a transition temperature which is reproducible. What does it mean? What is the relation of the transition temperature that has been determined to an actual structure? As an approach to defining the test, have the authors tried correlating it to the drop-weight test?

MR. CARL E. HARTBOWER (*author*).—With regard to Mr. Brandt's request that I amplify my statement that we believe the low-blow transition temperature to be significant, consider definitions based on an arbitrarily selected energy level. Although ship-fracture studies have indicated that a 10 or 15 ft-lb energy level is significant to service involving rimmed or semikilled steel, other investigations, particularly those of Mr. Pellini and his associates at the Naval Research Laboratory, have shown that full-killed and low-alloy high strength steels require a higher energy level at a given service temperature to be safe. In other words, a fixed energy level is an arbitrary criterion. With the low-blow technique, on the other hand, the crack initiation and crack propagation stages of fracture are separated. When the initial low blow is struck, a ductile crack is initiated. Adiabatic temperature-rise effects associated with the attendant plastic deformation are avoided by recooling the specimen prior to the second (crack propagating) blow. Then, upon delivering the subsequent blow, cracking is reinitiated and propagated as cleavage with very little plastic deformation at and below the transition temperature. Thus, the low-blow transition temperature is believed to be the maximum temperature at which self-propagating (catastrophic) cracking can occur.

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[1] Irradiations Engineer, General Electric Vallecitos Atomic Laboratory, Pleasanton, Calif.

With regard to Mr. Brandt's question as to the relation between the low-blow transition temperature and performance of actual structures, this, of course, is the goal of every investigator, to correlate his test with service. We hope to establish such a correlation in the near future, using materials from casualty ships, fractured pressure vessels, or any structural materials that have a history of service failure. We believe, based on the fundamental nature of the low-blow concept, that the test will correlate with service.

Mr. Brandt inquired as to whether we have attempted correlation with the Naval Research Laboratory drop-weight test. We have not; however, we believe that the low-blow transition temperature and the nil-ductility transition (NDT) as obtained from the drop-weight test will correlate, provided the weld heat-affected zone in the drop-weight specimen does not arrest the cleavage initiated in the crack-starter weld. Stout and his associates at Lehigh have published some test results showing that the crack-starter heat-affected zone may have a marked effect on the NDT temperature in some steels. In the low-blow test there is no weld heat-affected zone; therefore, correlation can be expected only in those steels that do not develop a notch-tough heat-affected-zone microstructure.

MR. C. MYLONAS.[2]—I wish to make a few remarks, without by any means wishing to minimize the value of the test presented here, which I think is basic. The tests seem to me of great interest because they produce the initiation of fracture from a very sharp crack. My first point is that, if I am not mistaken, the charts shown (see Fig. 1 of paper) show a good correspondence of the temperature transition range of the transition curves with those of the Charpy V-notch specimen, but less so with the keyhole. Often transition temperature is defined by an energy value, but it may be more realistic to speak of a transition range extending between the temperatures of toe and heel of the transition curve.

The second point is that the independence of the results from crack depth does not seem to lend any support to the theories of fracture based on energy criteria since they predict that the average stress at fracture should be inversely proportional to the square root of the crack length.

The third point concerns the interpretation of the results, in particular about the separation of fracture initiation on the first blow and fracture propagation on the second. To my mind a crack has to be reinitiated every time it stops. Propagation of a crack is associated with high speed of fracturing and the corresponding dynamic conditions which are completely different from the conditions prevailing during initiation.

From Fig. 2 of the paper, there seemed to be a lot of plastic deformation on the remainder of the section. Did any of it happen during the second blow? With reference to that deformation, the remainder of the section was not all in a virgin state. Some part of it, among others the root of the notch, must have been prestrained by the first blow.

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[2] Associate Professor of Engineering, Brown University, Providence, R. I.

MR. HARTBOWER.—With regard to Mr. Mylonas' first point, it is true that the temperature range corresponding to the transition from high- to low-energy values is approximately the same for the V-notch Charpy specimen when obtained by the conventional method and by the low-blow method. However, for most practical purposes, the engineer wants a specific temperature; to get such a temperature from conventionally obtained transition curves, an arbitrary energy level must be used in defining transition temperature. With the low-blow technique the inflection in the energy-to-propagate transition curve gives a definite and meaningful temperature.

With regard to the extent of the plastic deformation associated with the *second* blow, a partial answer can be obtained by examining a series of specimens tested over a range of temperature encompassing the low-blow transition. As the temperature is lowered, there is less and less evidence of deformation in the test section. Directly under the notch, there is always evidence of plastic deformation; that is, the initiating crack is always ductile. At and below the transition temperature, the only evidence of ductility in the fracture surface is shear at the biaxially stressed surfaces. Thus, energy-to-propagate values at temperatures below the inflection in the transition curve represent, in large part, the energy to fracture at the free surfaces.

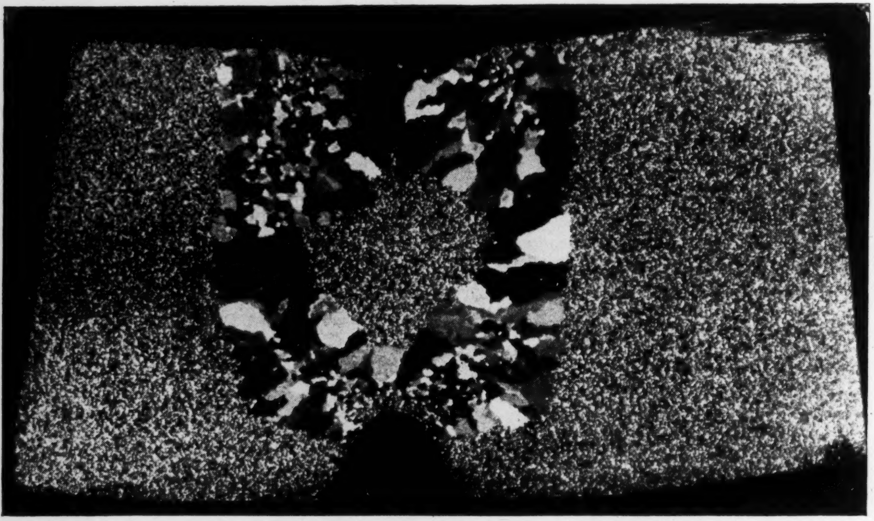

FIG. 8.—Strain Distribution in a V-Notch Charpy Bar After Impact With a 30 ft-lb Low Blow.

With regard to the extent of plastic deformation produced by the *initial* low blow, the accompanying Fig. 8 shows the distribution of strain in a Charpy specimen struck a 30-ft-lb low blow. The material was an 0.04 per cent carbon steel annealed for 45 hr at 850 C after the low blow to promote abnormal grain growth in the plastically deformed areas.

The specimen was subsequently sectioned, polished, and etched. Note that the central area was subjected to little or no plastic deformation by the 30-ft-lb blow. With a lesser blow, the undeformed central area would be larger; in fact there would be no abnormal grain growth at all in the section about the neutral axis.

MR. GEORGE M. ORNER (*author*).—One of Mr. Mylonas' questions dealt with the energy required to "reinitiate" the crack when the second, or crack-propagating blow was struck. I assume that Mr. Mylonas had in mind the experiments of Felbeck and Orowan where they demonstrated that the reinitiation of a *brittle* crack was always preceded by copious plastic deformation and the formation of a narrow band of fibrous or shear cracking. It appears that at the extremely high strain rates associated with brittle crack propagation, the micro deformation occurring at the tip of the advancing crack is sufficient to produce the triaxial tension necessary to maintain cleavage stress levels, but that at the low strain rates during reinitiation, much larger volumes of material must be deformed for this purpose. In the low-blow test, the "initiating cracks" are ductile cracks, and the volume of deformed metal associated with the ductile crack is sufficiently large so that when the second (crack-propagating) blow is struck, triaxial tension initiates cleavage with very little, if any, additional plastic deformation. We believe that the triaxial tension resulting from the low-blow-induced deformation contributes far more to the subsequent initiation of cleavage than the crack itself.

Regarding the deformation (lateral expansion) noted on the compression faces of the specimens fractured at low temperatures, this deformation, or by far the greater part of it, occurred as a result of the crack initiating blow. The small amount of deformation that occurs during crack propagation at temperatures below the transition is, we believe, the result of the work necessary to propagate the crack in the biaxially-stressed areas adjacent to the surfaces, that is, in producing shear lips. In our earlier work we plotted lateral-expansion *versus* temperature to obtain the low-blow transition, instead of plotting energy *versus* temperature. In these curves you can see exactly how much of the expansion occurred as a result of the propagating blow. This work was published in the December 1957 issue of *The Welding Journal*, p. 521-s.

MR. S. L. HOYT[3] (*by letter*).—I believe the results are capable of another interpretation to which I would like to call the authors' attention.

The first, or low blow, impact alters the specimen geometry and the standard impact is then made on a bar with a sharp notch. In other words, the final tests are made with a different notched bar and its effect on the behavior (energy absorption or ductile *versus* brittle break) or on the transition curve can be no more fundamental than with a test of any other notch geometry.

The author's technique is very similar to that used by Pellini at the Naval Research Laboratory when he deposits a brittle weld bead on his test specimen, and to that of the Notched Weld Bead Bend Test. The first deformation opens up a sharp crack from which point the behavior of the specimen depends on its response to the propagation of that crack. In other words, my interpretation is that the authors, instead of testing steel with a standard V or Keyhole notch, are simply using another notched bar with an initial crack. A large amount of past testing has shown that tests with different notches give different results, with no correlation between them. As opposed to this is the finding that after the low blow is given, the test bars give the same result. This will be considered subsequently.

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[3] Metallurgical Consultant, Battelle Memorial Inst., Columbus, Ohio.

It seems to me that the only way to develop something fundamental must be to establish the relationship between the controlling properties and the behavior under the stress system to which the steel is subjected. Zaustin (**1**)[4] has recently discussed notch behavior, whether ductile or brittle, from this standpoint and he points out that it depends on the ratio of the cohesive strength to the shear strength and upon the ratio of the normal stress to the shear stress. In 1938 (**2**) I presented the same doctrine and developed the concept of the "match point" where the two ratios are equal. More recently (**3**) I showed how the shift in the match point as the temperature and rate of deformation vary, or in the change from ductile to brittle behavior, was to be understood in terms of the effects of those variables on the shear strength. That is for a specific notch or stress geometry. If there is a change in the notch, that introduces a new stress system with a new stress ratio, that, of course, changes the match point and the transition curve. Incidentally, this approach also clarifies the effects of temperature and strain rate. Both here and abroad some have held that they are equivalent effects and can be quantitatively related by a constant factor. It is now clear that each exerts its effect individually by the way it changes the shear strength. The results of the low blow test should be viewed in this light.

I think that what the authors accomplished is the determination of the ability of the steel to resist crack or fracture propagation. With the same notch (the crack which is produced by the low blow), it cannot be surprising that the temperature of the match points at about −80 C in Fig. 1 of the paper is not influenced by the original machined notch. Incidentally this phase of fracture propagation is complex and appears to require more detailed examination such as that of the Irwin, Kies, and Smith paper (**4**).

Finally, it is fundamental in the service performance of steel to understand the influence of notch geometry on fracture initiation, not just the propagation phase however important the latter may be. In numerous applications, and for economic reasons, steel must be used at temperatures below the match point for the sharp crack. The sole recourse is that of avoiding notches which will initiate the brittle and self-propagating fracture. I have recently cited a case of steel which is used down to −30 F (**5**). An easy metallurgical solution would have been the use of a normalized, fine-grain steel with a favorable analysis, but the cost prohibited it. The correct solution under the circumstances was the use of a well-made, though cheaper, semikilled steel along with improved design and good fabrication and inspection technique.

MESSRS. HARTBOWER AND ORNER (*author's closure*).—We would like to stress that the low-blow method of impact testing is specifically designed to study the ability of steel to resist fracture propagation. The low-blow transition temperature is the maximum temperature at which the energy required for crack propagation is less than the elastic energy released as a result of the crack's own extension. Such a crack is "self-propagating" and of the catastrophic type observed in steel structures. In terms of the "match" point, the low-blow transition temperature is the highest temperature at which the match point is reached under the specific conditions existing at the tip of an *advancing* cleavage crack in the undeformed material about the neutral axis of the Charpy specimen. We believe that, in the center of a Charpy specimen, the conditions of restraint and strain rate at the tip of a dynamic cleavage crack are independent of specimen geometry and, in fact, all test variables except temperature. The maximum temperature at which cleavage cracking is initiated (that is, the temperature at the match point for the deformed material just below the notch) is of no consequence to the low-blow transition temperature determination except that cleavage must, of course, be initiated at a temperature at least as high as the low-blow transition.

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[4] The boldface numbers in parentheses refer to the list of references appended to this discussion, see p. 639.

As you suggest, the low-blow test is very similar to the NRL drop-weight test, except that it is devoid of the complicating effects of the heat-affected zone which in some steels may seriously depress the observed transition. In either test the stressed material is subjected to a propagating cleavage crack, thus duplicating the conditions at the tip of a catastrophically propagating crack in a service structure. In the low-blow test the crack propagates either by elastic energy with the data plotting at or below the inflection, or with difficulty requiring additional plastic-deformation work for its extension with the data plotting above the inflection.

We agree that for many service structures it is uneconomical to use a material that is capable of resisting brittle crack propagation at the lowest anticipated service temperature. In such cases, the only recourse is to eliminate as many notches as possible and hope that brittle cracking does not initiate from an overlooked defect or from subsequent damage. However, we believe that structural integrity can be guaranteed only if the material used for the structure is capable of resisting brittle-crack propagation at the lowest anticipated service temperature.

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FRACTURE STRENGTHS RELATIVE TO ONSET AND ARREST OF CRACK PROPAGATION*

BY G. R. IRWIN,[1] J. A. KIES,[1] AND H. L. SMITH[1]

SYNOPSIS

The conditions of stress which control spreading of a deeply embedded tensile crack are essentially those of plane strain. If the thickness of a plate containing a through crack is small enough, crack extension is accompanied by substantial amounts of plastic thickness reduction at the leading edge of the crack. A generalized plane-stress viewpoint is then applicable, and a relatively tough shear mode of fracture occurs. Methods have been developed for duplicating both the plane-strain and plane-stress conditions in laboratory measurements of fracture strength $\mathcal{G}_c$.

Observations of fracture mode, coupled with $\mathcal{G}_c$ measurements, suggest that it may be possible to predict which fracture mode will occur in terms of the plate thickness, yield stress, and the value of $\mathcal{G}_c$ for completely plane strain fracturing. The relationship thus outlined appears to be in agreement with the observed ductile-brittle fracture transition of low-carbon structural steel with reduction of temperature or with increase of stress loading speed.

Measurements of $\mathcal{G}_c$, coupled with stress analysis and estimates of starting crack size, assist the analysis of important problems—for example, fracture of pressurized fuselage of commercial jet aircraft, bursting of large steam turbine-generator rotors, structural vulnerability of combat airplanes, shattering of transparent plastic canopies, and crack propagation in rocket engine vessels.

Conventional procedures for prevention of fracture failures are qualitative rather than quantitative. An approximate but quantitative estimate of failure by general yielding may be made by comparison of net-section stress to yield stress. However, a safety factor must then be used to cover possible errors of this estimate and the chance of fracture failure as well.

It may be assumed that all fractures start from flaws, that cracks develop from flaws at various locations and time rates, depending upon local conditions, and that rapid extension of the most serious of these cracks initiates terminal fracture failure. If the consideration given to flaws of greatest weakening influence is limited to their statistical effects upon strength and strength fluctuations, then the calculated load-bearing capacity must be regarded as equally uncertain. In this discussion, it is assumed that careful study of a proposed structure would suggest estimates of the position and length of the largest cracks which might develop from flaws and stress cycling or which might be overlooked in inspection. A quantitative estimate of load-bearing capacity then becomes possible, based upon analysis of conditions for rapid extension of these largest cracks (1).[2] The measurement procedures which will be discussed in this paper are those which provide information needed for quantitative strength calculations of this kind.

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* Presented at the Sixty-first Annual Meeting of the Society, June 22–27, 1958.

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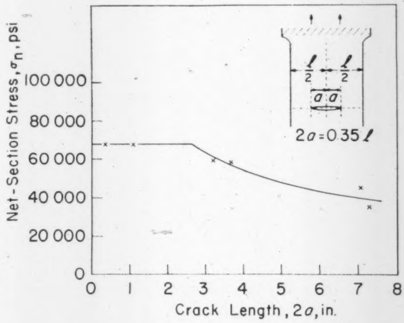

FIG. 1.—Net-Section Stress at Instability *Versus* Crack Length for 7075-T6. Face supports suppressed buckling across the span of the crack; plate thickness constant at 0.032 in.

Figure 1 shows the results of various tests in which a central slot with jeweler's hack saw extensions of the ends is used to represent a crack. For the tests shown, the applied load was simple tension normal to the plane of the crack-like flaw. The ordinate is average tensile stress on the net section. The abscissa is crack length. The size of the starting crack, as represented by the central slot, was kept in proportion to the specimen size.

Two regimes of behavior are evident. For small abscissa values, the net-section stress changes very little. Beyond a certain size, the net-section stress decreases in proportion to the inverse square root of the starting crack length. A behavior of the latter type, suggesting applicability of a Griffith-type viewpoint toward fracture strength, has often been noted (**2,3,4,5,6**).

Consider for the moment that the test samples represented in Fig. 1 are the structures whose strength is to be estimated and ask what rules should be followed. It must be kept in mind that a certain amount of plastic deformation generally accompanies crack extension in engineering materials (**7,8,9**). When this plastic deformation extends across the net section prior to fracture propagation, the supportable load can scarcely be less than the product of section area times the tensile yield stress. In this regime of behavior, the net-section stress capable of support changes little with crack length, because the net section acts like a plastic hinge or link, limiting the average stress to the yield stress – tensile stress range (**10**).

However, as the starting crack increases in size, the plastic strain zone does not increase in proportion (**8,11**). Thus a second regime of behavior exists in which the plastic strains are limited, say, to less than half the span of the net section. In this regime, the magnitude of the net-section average stress decreases toward zero with an increase of the abscissa in Fig. 1.

The nature of the reduction of net-section stress with increase of crack length in the second regime suggests that the supportable load could be calculated, neglecting plastic strains, since either a modified Griffith theory (**8,12**) or a notch stress concentration viewpoint (**13,14**) would predict the observed proportionality of net-section stress to inverse square root of crack length. It is apparent that an elementary approach to prediction of the tensile failure of the model structure just discussed requires two complementary viewpoints: (1) the extension of plastic deformation across the net section, neglecting crack growth, and (2) the extension of a crack across the net section, neglecting plastic deformation. We are primarily concerned with the second viewpoint. To estimate the tensile load necessary to cause fracture propagation, we need strength measurements by means of which the crack extension process can be related to the surrounding stress field.

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[2] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 656.

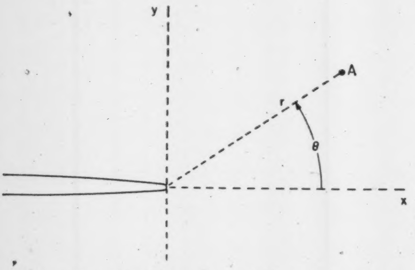

FIG. 2.—Two-Dimensional Crack Showing x, y, r, and θ Coordinates.

GENERAL REQUIREMENTS FOR THE TESTS

The nature and requirements of strength measurements can be understood in terms of the stress field near the end of a crack. In the elementary view adopted here, at first only the stress field is considered, neglecting plastic strains. Equivalently, one may assume that the plastic strains are confined to a small fraction both of the crack length and of the unbroken net section. The establishment of this situation experimentally is assisted by the fact that during crack growth the region of large plastic strains close to the leading edge of a crack does not expand in proportion to the crack length.

Figure 2 shows a system of the rectangular axes X and Y and a system of the polar coordinates r and θ at the leading edge of an internal free surface representing a crack. A Z-axis perpendicular to the plane of the figure is not shown. If we assume that the resolved shear stresses on the plane of expected crack extension are zero, then for a point A close to the crack edge the stresses are given by the following equations taken from reference (**4**):

$$\sigma_y = \mathcal{K}\,\frac{\cos\frac{\theta}{2}}{\sqrt{2r}}\left(1 + \sin\frac{\theta}{2}\sin\frac{3\theta}{2}\right) \ldots\ldots\ldots (1)$$

$$\sigma_x = \mathcal{K}\,\frac{\cos\frac{\theta}{2}}{\sqrt{2r}}\left(1 - \sin\frac{\theta}{2}\sin\frac{3\theta}{2}\right) - \sigma_{ox} \ldots (2)$$

$$\tau_{xy} = \mathcal{K}\,\frac{\sin\frac{\theta}{2}}{\sqrt{2r}}\cos\frac{\theta}{2}\cos\frac{3\theta}{2} \ldots\ldots\ldots\ldots\ldots (3)$$

$$\sigma_z = \nu(\sigma_y + \sigma_x), \text{ (plane strain)} \ldots\ldots\ldots\ldots (4)$$

$$\sigma_z = 0, \text{ (plane stress)} \ldots\ldots\ldots\ldots\ldots\ldots (5)$$

In these equations, the coordinates r and θ are as shown in Fig. 2, and ν is Poisson's ratio. There are only two parameters, $\mathcal{K}$, and σ_{ox}, through which the stress field near the leading edge of the crack is related to applied loads, crack length, and test structure shape. Experiments, for example those reported in this paper, have shown that the crack extension process is quite sensitive to a plane stress to plane strain shift in the stress environment. However, the amount of uniform stress parallel to the crack surface represented by the parameter σ_{ox} is of minor importance. This has been confirmed by a limited number of trials.[3] Thus the crack extension process is primarily controlled by the value of $\mathcal{K}$ and by the effective degree of plane strain.

The selection of the stress intensity factor $\mathcal{K}$ as the dominating stress field parameter is closely related to two well-known concepts of fracture strength: the Griffith crack theory (**15**) and the Neuber plastic particle concept (**13**). Regarding the Griffith theory, it has been shown (**16**) that

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[3] Data pertaining to these trials are still incomplete.

$$\mathcal{G} = \frac{\pi \mathcal{K}^2}{E} (1 - \nu^2), \text{ (plane strain)} \ldots\ldots\ldots (6)$$

and

$$\mathcal{G} = \frac{\pi \mathcal{K}^2}{E}, \text{ (plane stress)}, \ldots\ldots\ldots (7)$$

where E is Young's modulus and $\mathcal{G}$ is the rate of release of strain energy with crack extension.

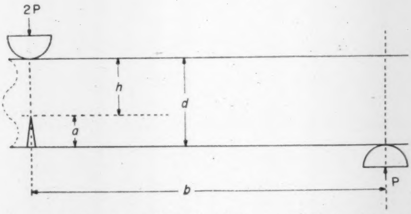

FIG. 3.—Right Half of Notched Bar Subjected to Bending by Forces Acting at Three Points.

In the Griffith theory, crack propagation was assumed to take place when the value of $\mathcal{G}$ became larger than twice the surface tension. In this paper, the basic assumption is more general. The parameter $\mathcal{G}$ is the force conjugate to time rate of crack extension (**4,17**). We assume only that the fracturing process is progressive and that the response in terms of speed of crack extension to an applied crack extension force $\mathcal{G}$ may be learned from experimental observation.

Consider next the Neuber plastic-particle concept (**13**). Neuber suggested that, after some plastic straining at the root of a sharp notch, the notch stress concentration factor might be calculated on the basis of an average value of the elastic theory stress through the deformed region, rather than on the basis of the maximum stress at the root of the notch. Applying this concept to a region of deformation beyond the edge of an internal free surface representing a crack, one finds from Eq 1, with θ equal to zero,

$$(\sigma_y) \text{ avg} = \frac{1}{\epsilon} \int_0^{\epsilon} \sigma_y \, dr = \mathcal{K} \sqrt{\frac{2}{\epsilon}} \ldots\ldots\ldots (8)$$

so that

$$\mathcal{K} = \sqrt{\frac{\epsilon}{2}} (\sigma_y) \text{ avg} \ldots\ldots\ldots (9)$$

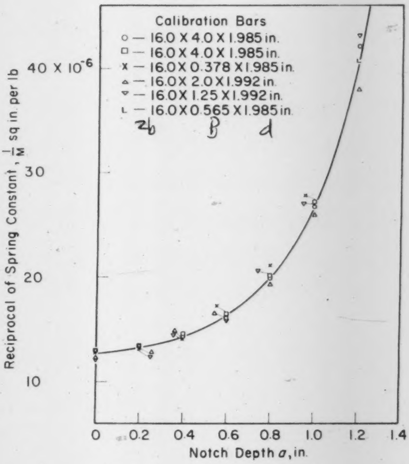

FIG. 4.—Reciprocal of Spring Constant, $1/M$, as a Function of Notch Depth a.

Here the deformed region, the Neuber plastic particle, is assumed to extend a distance ϵ beyond the edge of the internal free surface.

Another contact of the present analysis with the work of Neuber is obtained by noting that in all of Neuber's expressions for maximum stress σ_m at the notch root, the product of σ_m by the square root of notch root radius ρ approaches a finite non-zero value as ρ approaches zero. For a notch of very small flank angle, one may note that the relation of this limit to $\mathcal{K}$ is given by

$$\mathcal{K} = \lim_{\rho \to 0} \left(\frac{1}{2} \sigma_m \sqrt{\rho} \right) \quad \ldots\ldots (10)$$

The experimental investigation of crack propagation in terms of $\mathcal{K}$- or $\mathcal{G}$-values for practical strength calculation purposes is simplified by the fact that the speed of crack extension is quite sensitive to the magnitude of the applied crack extension force. It has been the authors' experience that the critical values $\mathcal{K}_c$ and $\mathcal{G}_c$ characterizing onset of rapid crack extension can be determined with a precision similar to that with which one can measure the critical yield stress characterizing onset of rapid plastic yielding.

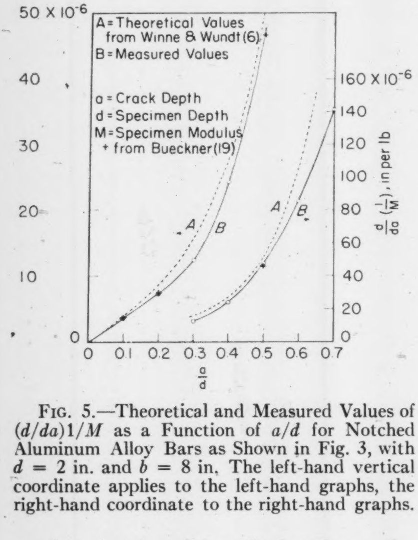

FIG. 5.—Theoretical and Measured Values of $(d/da)1/M$ as a Function of a/d for Notched Aluminum Alloy Bars as Shown in Fig. 3, with $d = 2$ in. and $b = 8$ in. The left-hand vertical coordinate applies to the left-hand graphs, the right-hand coordinate to the right-hand graphs.

The essential fracture strength measurement requirements from the preceding discussion are:

1. In order that the stress field environment of the advancing crack edge be approximately given by elastic theory equations, the test should be such that the average net-section stress is substantially less than the yield stress.
2. The test procedure must permit knowledge of $\mathcal{G}$ (or $\mathcal{K}$) as a function of load and crack length from theoretical or experimental stress analysis.
3. Crack length and load at onset of rapid fracturing must be observed or inferred from other measurements.

NOTCHED BEND TEST BARS

Figure 3 shows a notched bar under a bending load as used by the authors of this paper. In the closely related notched bar bend tests discussed by Lubahn and Yukawa (**18**) and by Winne and Wundt (**6**), the central load $2P$ in Fig. 3 is divided and applied at two points. Values of $\mathcal{G}_c$ were reported by Winne and Wundt, based upon a theoretical procedure (**19**) to determine the applicable stress analysis. In the present paper, an equivalent experimental procedure is used.

The relation of the load to the values of $\mathcal{G}$ was shown in reference (**1**) and (**20**) to have a simple form in terms of the change with crack depth of the spring constant per unit thickness. If the applied load per unit thickness is $2P$ and the elastic displacement of the position of load application is e, then

$$2P = Me, \quad \ldots\ldots (11)$$

where M is the spring constant per unit thickness. It was shown (**20**) that

$$\mathcal{G} = \frac{1}{2} (2P)^2 \frac{d}{da} \left(\frac{1}{M} \right) \quad \ldots\ldots (12)$$

Using calibration bars of 7075-T6 aluminum, measurements of $\frac{1}{M}$ were made for a series of notch depths a. The notch root was machined to a radius of about 0.008 in. Loads were held well below those needed for crack initiation at the root of the notch. The results are shown on Fig. 4. By curve fitting, values of the slope $\left(\frac{d}{da}\right)\frac{1}{M}$ as a function of a were obtained. These are shown as curves B on Fig. 5. Theoretical values of this function for an approximate stress analysis for notched bars under four-point loading were obtained from Fig. 16 of reference (6) and are shown for comparison as curves A in Fig. 5. Bueckner's theoretical estimates for three-point loading are also indicated. Although the experimental procedure based upon spring-constant measurements requires high precision, careful work can be expected to provide a trustworthy relationship between applied load and $\mathcal{G}$-value.

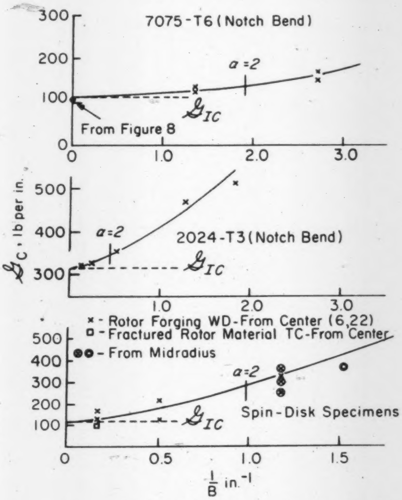

FIG. 6.—Critical Crack Extension Force Values as a Function of Reciprocal Plate Thickness.

Figure 6 shows results of measurements of $\mathcal{G}_c$ for two aluminum alloys and for steels cut from two large forgings. Except for the lower graph, the bend test procedure illustrated in Fig. 3 was employed. The results for $\mathcal{G}_c$ increase with the reciprocal of the test bar thickness B, as do the $\mathcal{G}_c$ values from reference (6) shown in the lower graph. As the thickness of the test bar is reduced, the zone of plastic strains at the sharp root of the notch receives less support from the surrounding elastic region and tends to expand, with a consequent increase of the fracture work rate $\mathcal{G}_c$. Only a small part of the possible increase of fracture toughness with decrease of test plate thickness is shown in Fig. 6.

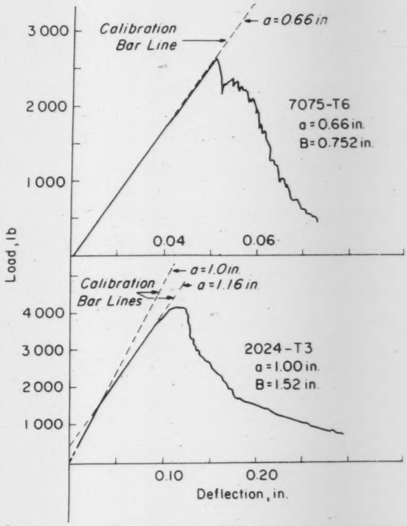

FIG. 7.—Typical Load Deflection Curves for 7075-T6 and 2024-T3 Aluminum Alloys During Tests of Specimens as Shown in Fig. 3.

Bend tests of the type shown are a convenient procedure for measuring $\mathcal{G}_c$ under the plane strain conditions associated with large test plate thickness.

Figure 7 shows two load-displacement records for typical bend tests. As loading proceeds, plastic strains occur at the root of the notch. In the regions of largest tensile stress, and with the aid of localized plastic strains, fracture origins grow and eventually join into a crack with a free surface connection to the notch root. Whether the plastic strain zone at onset of rapid fracture is small enough so that a stress analysis may be assumed applicable which neglects plastic strains can be judged by comparing the relative sizes of the elastic and inelastic portions of the load-displacement record. For the 7075-T6 record shown in Fig. 6, prior to the sharp load drop at first crack propagation, the amounts of inelastic displacement were small enough to permit straightforward determination of a $\mathcal{G}_c$ value. However, the amounts of slow crack development and plastic strain prior to crack propagation, particularly for the 2024-T3 test bar, were considered large enough to warrant a crack-depth correction procedure.

The $\mathcal{G}$ value characterizing the stress field surrounding the crack extension process is influenced not only by deepening of the crack from crack growth at the notch root but also by stress relaxation in the associated zone of plastic strains. One may observe in Fig. 6 that the spring constant or slope of the load-displacement record prior to crack extension was less for the 2024-T3 test bar than for a calibration bar with a relatively blunt notch of equal depth. Using the curve shown in Fig. 5, the effective crack depth a was found which corresponded to the apparent spring constant for large loads as indicated in Fig. 6. In the results for aluminum shown in Fig. 6, the $\mathcal{G}_c$ values were calculated using this effective or corrected notch depth. Without this correction, the results for 2024-T3 aluminum would have shown a less pronounced upward trend with decrease of bar thickness. A more accurate experimental determination of effective crack depth is possible using procedures such as those discussed by Trimble (21). The need for any plastic strain correction of the notch depth can be eliminated by increasing the test-specimen dimensions and notch depth.

The lower portion of Fig. 6 shows results for a special technique of notched specimen testing developed to assist the study of large steam turbine generator fractures. Measurements of $\mathcal{G}_c$ by spin tests of centrally notched disks were discussed in the Winne and Wundt paper (6). This paper, which presents results by a larger group of investigators, is of special importance in showing correct application of fracture mechanics principles to a serious fracture strength problem. The paper shows the correlation of spin-disk test results with notched-bend tests and with transition temperature. In addition, the interpretive and analytic comments assist and support the present discussion. The spin-disk test results shown in Fig. 6 were obtained using 24-in. diameter disks with a 3.25-in. central hole. These were sharply notched outward from the central hole. The speed of rotation necessary for fracture propagation was measured, and $\mathcal{G}_c$ was calculated with the aid of an appropriate stress analysis. The selected results are among the lowest in terms of fracture strength quality reported in the Winne and Wundt paper. They are not representative of current improved rotor steels. Detailed descriptions of these and other rotor steels may be found in reference (22). Three results for $\frac{3}{4}$-in. thick disks of 9-in. outer diameter are included, although in these tests net-section stress was more than 0.6 times the yield stress.

PLANE-STRAIN FRACTURE CONDITIONS

The conditions for fracturing under essentially plane-strain conditions as opposed to the shear fracturing of thin sheets can be approximately estimated by the following analysis. In terms of the coordinates r and θ of Eqs 1, 2, and 3, if the zone of plastic strains near the crack is confined to values of r somewhat less than the specimen thickness, a large measure of elastic support is present to

TABLE I.—RESULTS FROM TYPICAL FRACTURE-STRENGTH MEASUREMENTS.[a]

Material	Thickness, in.	Yield Stress, psi	$\mathcal{G}_c$ Measured, lb per in.	$\mathcal{G}_{Ic}$ Estimated, lb per in.	α	Method[c]	Fracture
Glass, lantern slide covers:							
moist	0.05	>100 000	0.04	0.04	>1000	A	Plane strain
2 per cent RH	0.05	>100 000	0.08	0.08	>500	A	Plane strain
Poly(methyl methacrylate):							
cast	0.25	~10 000	4	4	16	A	Plane strain
hot-stretched	0.25	~10 000	25 to 40	25 to 40	2.5 to 1.6	A	Plane strain
Ship steel, ABS:							
class C, reference (**27**), −40 C	1.00	100 000[b]	63 to 82	63 to 82	5.3 to 4.1	Test method of reference (**26**)	Plane strain cleavage
class C, 108 in. wide, 0 C	0.75	36 000	>1070	150	0.22	A	Plane stress shear
reference (**33**), −80 C	0.75	105 000[b]	~100	80	3.3	Residual Stress Zone Tests	Plane strain cleavage
Rotor Steel, nickel-molybdenum-vanadium, reference (**18**):							
quenched and drawn	6	101 000	960	<960	>2.1	C	Plane strain
pearlitic and upper-bainitic	6	89 000	135 to 180	135 to 180	9 to 12	C	Plane strain
SAE 4340	0.250	230 000	130 to 150	130	3.2	A, B	Plane strain
SAE 6150	0.075	260 000	275	225	0.75	A	50 per cent shear
2024-T3	0.032	47 000	~1500	300	0.02	A	Plane stress
	0.25	47 000	2800	300	0.18	A	Plane stress
	4	47 000	340	300	2.9	B	Plane strain
7075-T6	0.032	68 000	790	115	0.13	A	Plane stress
	0.750	68 000	134	115	3.0	B	Plane strain
	2-in. bar	67 000	115	115	..	D	Plane strain

[a] Measurements are at room temperature except as indicated.
[b] Dynamic values.
[c] A = Central slotted plate in tension.
B = Notched bend tests.
C = Spin-disk centrally notched.
D = Circumferentially notched circular bar.

restrict plastic contraction in the thickness direction, and the situation should be close to one of plane strain. McClintock's recent discussion of this point (**23**) may be noted. A rough estimate of plane-strain crack extension conditions with the aid of Eq 1 is as follows. Placing σ_y equal to the tensile yield stress σ_Y and θ equal to zero, the approximate radius r_Y to the edge of the plastic strain zone is given by

$$\sigma_Y = \frac{\mathcal{K}}{\sqrt{2r_Y}} \quad \text{..........(13)}$$

or

$$r_Y = \frac{\mathcal{K}^2}{2\sigma_Y{}^2}, \quad \text{...........(14)}$$

For onset of crack propagation,

$$\mathcal{K}^2 = \frac{E\mathcal{G}_{Ic}}{(1-\nu^2)\pi}, \quad \text{.........(15)}$$

where $\mathcal{G}_{Ic}$ is the critical crack extension force for plane-strain fracturing. Reduction by plastic strain of the plate thickness B is restricted, and a plane-strain situation exists when

$$B >> r_Y = \frac{E\mathcal{G}_{Ic}}{2(1-\nu^2)(\pi\sigma_Y{}^2)} \quad \text{....(16)}$$

If the thickness B is expressed as

$$B = \alpha\,\frac{E\mathcal{G}_{Ic}}{\sigma_Y{}^2}, \quad \text{...........(17)}$$

values of the relative plate thickness α furnish a rough guide for predicting whether a plate will break from a notch or starting crack with a low-ductility plane-strain fracture or with a shear fracture. With the value of α reaching or exceeding 2, the existing plane strain fracture conditions are accompanied by a relatively low value of $\mathcal{G}_c$, as can be seen from the limited available data discussed later in connection with Fig. 9 and Table I. If α is 0.2 or less, plate thickness reduction and shear fracturing occur, and the value of $\mathcal{G}_c$ is much greater. Mixed behavior is expected in the intermediate range of values.

For example, in the case of 1-in. thick ship steel at room temperature, taking $\mathcal{G}_{Ic}$ as 100 lb per in. and σ_Y as 36,000 psi, one finds α to be 0.43, an intermediate value. The result commonly observed in static notched-plate tension tests is that a plane-strain fracture starts from the central region of the notch, while shearing fractures begin at the plate surfaces. In this instance, plate thickness reduction develops, and the shearing mode of fracture prevails, leaving a segment of flat fracture at the notch. However, if the same fracture is started by impact, so that the crack forms rapidly, σ_Y is more than doubled, and α shifts to the range of 1.5 to 2.0. Then the plane-strain fracture mode persists, restricting the shear fracture to the plate surfaces.

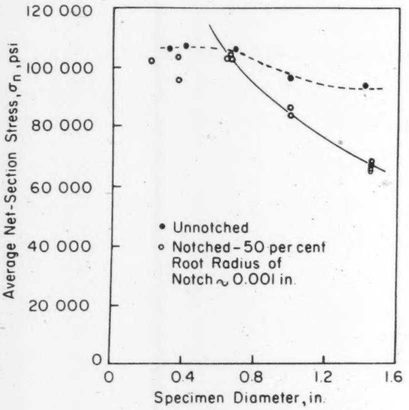

FIG. 8.—Effect of Specimen Size on the Strength of Notched and Unnotched Round Bars of 7075-T6 Aluminum in Tension, Reference (**29**).

CIRCUMFERENTIALLY NOTCHED ROUND TENSION BARS

Tension bars notched circumferentially to various depths have been used in a number of ways to determine mechanical strength in the presence of a notch. When the notch has a relatively small flank angle and sharp root radius, results of such tests can be employed to determine fracture strength for plane-strain conditions.

Figure 8 shows results obtained by Weiss and Klier (**24**) on specimens of 7075-T6 aluminum. The specimen axis had the direction of greatest extension during fabrication. The net-section area at the notch was half of the section area of the round tension bar. The data points in Fig. 8 show results of a fracture size effect study using different size test bars with the dimensions scaled. The graph of net-section stress σ_n, as a function of notch depth, reflects the characteristic features discussed in relation to Fig. 1. For the smallest specimens, yielding of the notched section limited the average net-section stress to approximately the net-section stress at fracture found with unnotched tension specimens. For the largest specimens, the trend of σ_n is one of proportionality to the inverse square root of the notch depth.

For these larger test specimens, the severing crack does not start uniformly all around the root of the notch. It forms first in a single weakest region near the notch root, then spreads from this origin across the notched section. A flat fracture traversing a thick plate similarly moves along by the forming and spreading of reinitiation cracks near the advancing edge. It is believed that the notch root radius for the test results in Fig. 8 was of such size that the onset of crack propagation for the largest test bars occurred under stress conditions similar to those controlling onset of crack propagation in the previously discussed bend tests of notched specimens.

In order to calculate the value of $\mathcal{G}_{Ic}$ corresponding to the results in Fig. 8, stress concentration factor curves published by Peterson (**25**) were employed. The approach to an infinitely small notch radius implied by Eq 10 was studied by calculating the product $\sigma_m\sqrt{\rho}$ for successively smaller values of ρ. It was concluded that for this test specimen[4] geometry

$$\mathcal{K} = 0.44\,\sigma_n\,\sqrt{D}, \qquad (18)$$

where D is the entire diameter of the test bar. With this equation and Eq 6, the curve through the plotted results for the large test bar was found to correspond to a value of $\mathcal{G}_{Ic}$ equal to 105 lb per in. The result is in fair agreement with the bend test values for $\mathcal{G}_c$ in Fig. 6.

CENTRALLY NOTCHED FLAT SHEETS UNDER TENSION

The requirement of having the average net section stress below the yield stress in many cases necessitates use of a length of net section which is ten or more times as large as the plate thickness. In this situation, the most convenient test specimen is usually a centrally notched sheet in tension, as shown in Fig. 1. If the length of the test section is as much as three times the width, one may choose between a variety of end grip procedures without influence on the test results. Wedge grips of the Templin type have been customary at the U. S. Naval Research Laboratory. In the case of $\frac{1}{4}$-in. plates of high yield strength steel, the specimen ends were made relatively wider than shown in Fig. 1 and the loads successfully transmitted through the shoulders. At the Naval Proving Grounds, tests of this nature have been conducted using a width to length ratio of 1 to 4 without shoulders. Loads were applied by two close-fitting pins in holes suitably located near the specimen ends.

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[4] An informal communication from H. F. Bueckner suggests that one should use 0.46 instead of 0.44 in Eq 18. A further correction due to an overlooked factor of 2 was noted by B. M. Wundt. Thus the numerical coefficient should be 0.23 instead of 0.44. A recalculation of $\mathcal{G}_c$ including a small allowance for depth of the starting crack beneath the notch suggests the $\mathcal{G}_c$ value of 80 lb per in.

In all of the tests discussed in this paper, satisfactory methods of preparation of the crack-simulating slot or notch must be considered. The practice which the authors recommend and attempt to follow in their measurements is one of providing a weakness to crack formation at the slot ends which exceeds the natural crack strength. This is done by providing notch sharpness sometimes coupled with local damage and precracking. A crack then starts and spreads slowly during the last stages of loading while the load is still increasing.

In all situations discussed here, $\mathcal{G}$ increases in proportion to the square of the applied tensile load. Excepting tests done with wedge forces, $\mathcal{G}$ also increases with crack length. Thus when slow crack growth occurs with increasing load, it may be inferred that the driving force $\mathcal{G}$, the resistance to crack extension, and the size of the plastic strain zone at the crack tip are all increasing more rapidly than the total crack length. Unstable fast crack extension develops after the growth of resistance to crack extension, that accompanies the increase in size of the crack tip plastic strain zone, begins to lag behind the growth of the crack-extension force $\mathcal{G}$. Ideally, this epoch occurs after the plastic strain zone at the crack tip has attained its full natural size. To insure that onset of fast fracture does not occur within the damage zone used for crack initiation, the size of this zone must be no more than a small fraction of the total crack length.

It has been found convenient to record the crack length and testing machine load simultaneously with a motion picture camera operating at 24 frames per sec. The crack length and load of the last frame before rapid crack extension are used in calculating the $\mathcal{G}_c$ value. As an alternative procedure, a drop of India ink may be placed in each end of the starting crack slot. The ink penetrates and stains the fracture surface while the crack spreads slowly and leaves the rapidly spreading portion of the fracture unstained. The maximum load and the crack length indicated by ink stain are then the recorded observations.

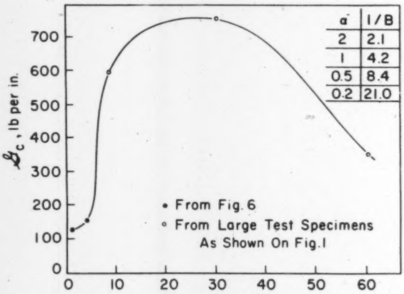

FIG. 9.—Critical Crack Extension Force Values as a Function of Reciprocal Plate Thickness for 7075-T6. Values of α shown in the upper right of the figure were calculated from Eq 17 using 72,000 psi for σ_Y and 115 lb per in. for $\mathcal{G}_{Ic}$.

If the crack length at onset of rapid fracture is not more than half the plate width, a stress analysis of sufficient accuracy for purposes of calculating $\mathcal{G}_c$ is provided by the Westergaard stress system for a repeated series of colinear cracks (16). The corresponding equation for $\mathcal{G}_c$ (plane stress) is

$$\mathcal{G}_c = \frac{l\sigma^2}{E} \tan \frac{\pi a}{l} \quad \ldots\ldots\ldots\ldots (19)$$

where l is the width of the test sheet, the length of the crack for onset of rapid fracture is $2a$, and σ is the average longitudinal tensile stress across the entire width of the test specimen.

Figure 9 shows measurements of $\mathcal{G}_c$ for a group of widely differing specimen thicknesses of 7075-T6 aluminum alloy. The figure shows the relatively abrupt increase of $\mathcal{G}_c$ with the change from plane-strain to plane-stress of the stress field near the crack. The subsequent drop of $\mathcal{G}_c$ with further decrease of plate thickness is believed to be due to a tendency of the plastic strain zone to extend away from the crack line an amount proportional to the plate thickness.

Although a substantial number of $\mathcal{G}_c$ values for flat sheets of high-strength alloy steel are in process of collection, only isolated groups of results are available at the present time. Table I shows a representative series of results on a variety of materials.

In the theoretical stress system pertaining to the centrally notched sheet in uniaxial tension, there is a compressive stress parallel to the length of the central slot which, at the borders of the slot, is equal in magnitude to the average longitudinal stress σ. When the width-to-thickness ratio is large enough, this compression will cause noticeable buckling across the span of the crack. Such buckling, when present, alters the stress system and adds a shearing motion to assist the separation process at the ends of the starting crack. A reciprocal square root relation between load and crack length has been found to apply in some instances in spite of the buckling effects. Tests of 0.032-in. thick sheets of the two aluminum alloys 7075-T6 and 2024-T3 under such conditions were made, and values thus obtained were lower by a factor of two than $\mathcal{G}_c$ values measured with buckling restrained by the use of lubricated face plates.

Where a sheet composes a wall of a pressure vessel, extension of a crack, whose length is large compared to the sheet thickness, is accompanied by considerable bulging at the crack borders, which again alters the stress system and adds a twisting action at the crack ends. Estimates of apparent $\mathcal{G}_c$ values based upon nominal membrane stress in situations of this type have given apparent $\mathcal{G}_c$ values more than a factor of ten less than the $\mathcal{G}_c$-value for flat sheet.

It is apparent that the arrest of crack growth in a pressure vessel wall requires maintaining or producing a flat-sheet situation, at least near the advancing edge of the crack. Local flat-sheet situations are produced, for example, by the circumferential stiffeners for crack arrest in commercial jet airplanes. The low apparent $\mathcal{G}_c$-values which pertain to various situations of pressure against one side or buckling are of interest for the understanding of fracture propagation in certain structures. However, for purposes of fracture strength analysis it is believed that the flat sheet values for $\mathcal{G}_c$ will usually apply to the critical situation of fracture initiation or fracture arrest. Thus restraint of sheet buckling is preferred in the $\mathcal{G}_c$ measurement procedure.

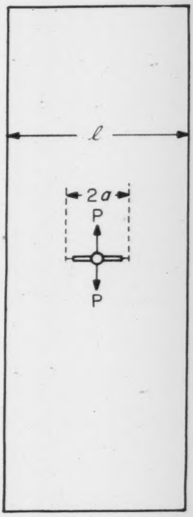

FIG. 10.—Wedge Force Specimen.

OTHER MEASUREMENT TECHNIQUES

When the material available for $\mathcal{G}_c$ evaluation does not have appropriate dimensions for one of the tests previously discussed, a procedure using wedge forces applied near the borders of the crack-simulating slot is sometimes applicable. Figure 10 shows a sample design for such a test procedure. The value of $\mathcal{G}$, when the wedge or splitting force is P per unit thickness, may be shown (16) to be:

$$\mathcal{G} = \frac{2P^2}{El \sin \frac{2\pi a}{l}}, \ldots\ldots\ldots (20)$$

where the crack length $2a$ is assumed to be not greater than $\frac{l}{2}$. In this test procedure, extension of the crack with the load maintained causes a reduction of the crack-end stress field, and the crack extension process is self-arresting. The value of the load to maintain a fixed velocity of crack extension increases with crack length until half the width of the test plate has been severed. Depending upon the strain-rate sensitivity of a material, the values of $\mathcal{G}$ necessary to maintain crack motion tend to be less than the $\mathcal{G}_c$ determined as discussed previously. This is due to the slower motion of the crack. The forces P shown in Fig. 10 may be applied either by a system of wedges calibrated for friction or by an opposing pair of hooks in tension.

By use of an experimental stress analysis procedure to establish the relation of the applied load and crack length to the $\mathcal{G}$ or $\mathcal{K}$ values, a large variety of fracture tests for measurement of $\mathcal{G}_c$ or $\mathcal{K}_c$ can be developed.

EFFECTS OF TEMPERATURE, THICKNESS, AND DYNAMIC LOADING

In a bend test study at the University of North Carolina on notched bars of 2024-T3 aluminum, Trimble (21) was able to estimate critical $\mathcal{G}$-values pertaining to arrest as well as to onset of rapid fracturing. The values found for these two conditions did not differ significantly.

High-strength aluminum alloys and very hard steels have relatively small dynamic increase of yield stress. For such materials, one would expect the arrest and start of rapid fracturing to occur at about equal conditions of the surrounding stress field. However, for steels in a softer condition, and for plastics generally, stress conditions for onset and arrest of rapid fracture may differ. For this reason it would be helpful to have a convenient measurement procedure for $\mathcal{G}_c$ determinations under conditions of fracture arrest. It would appear that additional test development work is needed on such a procedure. The nearest published approach to a fracture arrest measurement procedure is a test using impact application of wedge forces introduced by Feely and others (26). This technique and some derived estimates of fracture arrest $\mathcal{G}_c$ values for 1-in. ship plate steel were discussed by Wells (27). Essentially, an edge crack in a plate subjected to longitudinal tension is given rapid motion by impact application of wedge forces at the side of the test plate. For large tension loads, the started crack severs the plate. For sufficiently small longitudinal tension, the crack extension force, as a function of crack length, has a minimum which can be adjusted so as to produce fracture arrest. Although the test endeavors to keep the applied wedge force constant, it is not measured. The conditions of the test have, however, permitted approximate estimates of the critical crack extension force for arrest of rapid fracturing, as noted in the preceding discussion. These estimates, 62 and 83 lb per in. for 1-in. plates of ship steel at −40 C, may be compared to values in the range from 100 to 200 lb per in. for the plane strain $\mathcal{G}_c$ of pearlitic and upper bainitic steels of higher alloy content measured under static load conditions at room temperature.

Figure 11 shows the increase of plane-strain $\mathcal{G}_c$ values with temperature as reported by Winne and Wundt (**6**). Parker (**28**) has observed that the individual facets of a flat fracture in steel, while often showing a mixed behavior, tend to change from surfaces of shear type to those of cleavage type with lowering of temperature. It is not clear at present how much of the increase of fracture work rate as shown on Fig. 11 is due to growth in size of the shear lip regions at the free surfaces and how much is due to change in fracture type of the individual facets of the fracture in the embedded plane-strain regions. To illustrate the influence of a lowering of the transition temperature, one may note in Table I that the nickel-molybdenum-vanadium rotor steel when in a tempered martensite condition had a room temperature $\mathcal{G}_c$ value of 960 lb per in.

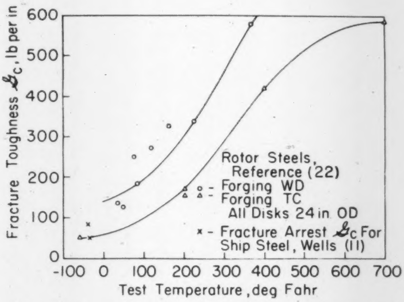

FIG. 11.—Variation with Temperature of $\mathcal{G}_c$ from Disk Bursting Tests, Reference (**6**).

Estimates by Wells of fracture arrest $\mathcal{G}_c$ values for ship plate steel are shown in Fig. 11. Unfortunately, $\mathcal{G}_c$ values for onset of plane-strain fracturing for ship plate steel are not available. Nor do we know fracture arrest $\mathcal{G}_c$ values for the rotor steels. Thus a direct comparison of $\mathcal{G}_c$ for onset and arrest of plane-strain fractures cannot be made. However, it seems unlikely that the values of $\mathcal{G}_c$ for onset of plane-strain fracturing in the ship steel would greatly exceed those for the rotor steels shown on Fig. 11. Considerations of this nature suggest that $\mathcal{G}_c$ for onset and arrest of rapid crack extension do not differ greatly when both are measured under plane-strain fracture conditions.

For steels, the convenience with which brittle fractures are produced in notched-bar impact tests by lowering the temperature may have resulted in a reduced awareness of brittle-fracture size effects. The transition from ductile to brittle fracture of sharply notched steel bars with increase of specimen dimensions received wide notice due to tests conducted by Docherty (**29,30**), who considered this as equivalent to a temperature shift. S. L. Hoyt (**31,32**) has advocated the use of this dimensional effect in studying the notched-bar impact response of some steels. He showed that double-width Charpy bars for some steels break in a brittle manner, although the single-width bars break in a tough manner. Although fracture mechanics analysis procedures can be applied to assist the understanding and correlation of various transition-temperature test methods, such a discussion is beyond the intended scope of this paper. Here it will merely be emphasized that the fracture strength of a steel should be considered for the entire range of section thickness that is of interest at a given temperature as well as for the entire range of temperature that is of interest for a given section thickness.

STRENGTH ANALYSIS APPLICATIONS

The customary remedies for structural failures due to crack propagation are of three kinds: (1) structural design improvements to reduce stress concentrations in some components or to provide for fracture arrest, (2) reduction of flaws, especially those embedded in a zone of residual stress, and (3) use of materials of greater fracture toughness. Combinations of these three remedies have solved many fracture strength problems. The practical importance of analysis by the methods of fracture mechanics appears when it becomes essential to know how much of each remedy is needed in quantitative terms or when one attempts to link together prior estimates of stresses, crack sizes, and material toughness so as to calculate in advance a service load which will be safe relative to fracture propagation.

A procedure for designs safe against fracture failure was discussed by Irwin and Kies (1), as noted in the introduction. It was suggested that experience accompanied by studies of stress distribution in a structure would suggest the size of the largest cracks likely to be formed in service or overlooked in inspection. When these estimates have been made, values of $\mathcal{G}$ for these cracks can be compared to measured values of $\mathcal{G}_c$ for the material. The upper limits of stress in service can then be estimated which will leave some margin of safety against fracture propagation. Suppose, for example, that a calculation of this type indicates likelihood of failure by fracturing at a nominal stress well below the yield stress of the material. Then if by using a material of lower yield strength an increase of fracture strength $\mathcal{G}_c$ is obtained, a substitution of this kind might well result in a structure capable of bearing larger stresses.

Measurements of $\mathcal{G}_c$ using spin-disk tests of centrally notched specimens as well as notched bars in bending were found to be helpful in the analysis of sudden fractures of large steam-turbine generator rotors. This application is noted here without further discussion because it was presented in some detail in the recent paper by Winne and Wundt (6).

Assume that the structure one has in mind is a pressure vessel and that bending moment superimposed upon high membrane stress may in local regions cause a crack to extend from a flaw in the high-tension surface through the wall thickness B. Assume the initial flaw at most would result in a through crack of length $2B$. Then to suppress crack propagation in the wall, it would appear sufficient to have a fracture toughness in the wall material corresponding to

$$\mathcal{G}_c = \frac{\pi B \sigma_Y{}^2}{E}, \qquad (21)$$

where $\mathcal{G}_c$ is the critical value for fracture arrest. Thus a crack of length $2B$, if developed, would be arrested and would, in fact, be unlikely to develop in a carefully fabricated component except from initial flaws of improbable largeness.

Looking at this situation from another viewpoint, if fracture tests of the wall plating show the fracture runs with a shear mode of separation, then one anticipates that

$$\alpha = \frac{B \sigma_Y{}^2}{E \mathcal{G}_{Ic}} < \frac{1}{4} \qquad (22)$$

$$\mathcal{G}_c > \mathcal{G}_{Ic} > \frac{4B \sigma_Y{}^2}{E} \qquad (23)$$

Thus $\mathcal{G}_c$ is larger than needed for the requirement represented by Eq 21.

Direct applicability of a strength analysis through fracture mechanics exists in the case of crack-arresting strips or frames for pressurized cabin aircraft. In this instance, it is desired that, if one frame and the adjoining sections of fuselage skin are sharply cut or cracked, crack arrest strips or the next frames will stop further spread of the fracture. Thus the crack length and membrane stress are known. As indicated by the preceding discussion, the reinforcing element must produce an approximate flat-sheet situation in the local region of crack arrest. With this assured, if the crack extension force for the given crack length x and membrane stress σ, as computed from

$$\mathcal{G} = \frac{\pi\sigma^2 x}{2E} \qquad (24)$$

is smaller than the flat-sheet $\mathcal{G}_c$ value, then successful arrest of the crack can be predicted. If the reverse is true, then the influence of the reinforcement strip or frame in reducing the stress field near the crack must be estimated. Although this calculation can be made only approximately, it is nevertheless useful in suggesting the magnitude of adhesive or rivet forces developed and their probable result in accomplishing arrest of the crack.

Investigation of the causes of all kinds of fracture failures after their occurrence are assisted by $\mathcal{G}_c$ fracture strength values for materials. Frequently the size of the starting crack can be found by study of the fracture. When the $\mathcal{G}_c$-value is also known, the tensile stress which acted across the fracture origin can be determined and consideration given to how this stress value developed. When the tensile stress in a region enclosing the starting crack is known, the apparent $\mathcal{G}_c$ value can be calculated and compared with fracture strengths considered normal for the material.

A variety of other applications of the fracture-mechanics viewpoint of this paper have been made—for example, the studies of fracturing from flaws in welded steel plates by Wells (**10**), by Carpenter and Linsenmeyer (**33**), and by Sopher, Lowe, and Rieppel (**34**).

SUMMARY

In view of the character and size of flaws in real structures, the fracture strength of a structural component is considered to be determined by the conditions for extension of an already started crack. Analysis of the elastic stresses near the edge of an internal free surface normal to the largest tensile stress suggests that the stress conditions controlling extension of a tensile crack can be described by the Griffith theory strain-energy-release rate $\mathcal{G}$ and by the degree of approach to a situation of plane strain. From measurements of the critical value $\mathcal{G}_c$ for onset of rapid crack extension, together with knowledge of the size and location of an actual or assumed starting crack, stress analysis procedures can be used to predict the loads necessary to cause fracture failure of a structure. The analysis is valid when the expected average net-section stress at the plane containing the crack is less than the yield stress.

Values of $\mathcal{G}_c$ may be determined as a function of section size, temperature, and yield stress using, for example, centrally slotted plates oı sheets in tension, notched bars in bending, or circumferentially notched round bars in tension. The ends of the cut representing the starting crack should be sufficiently sharp or locally damaged so that an appreciable slow extension of the crack precedes onset of rapid fracturing.

A large and relatively abrupt decrease of $\mathcal{G}_c$ occurs with increase of plate thickness when the plate thickness becomes substantially larger than the span of the plastic strain zone beyond the leading edge of the crack. The change in fracture toughness is accompanied by a change from a shear fracture to a flat tensile fracture and corresponds to the change in the governing stress conditions from plane stress to plane strain. The plate thickness necessary for such a fracture mode transition can be estimated in terms of a relative plate thickness parameter α given by

$$\alpha = \frac{B\sigma_Y^2}{E\mathcal{G}_{Ic}}$$

where B is the plate thickness, σ_Y is the yield stress, E is Young's modulus, and $\mathcal{G}_{Ic}$ is the $\mathcal{G}_c$ value for plane-strain fracturing. From available data, an α value greater than 2 ensures a mode of fracture which is primarily of the plane-strain type. A plane-stress fracture or shear fracture develops when α is less than 0.2.

When accompanied by appropriate analyses of stress conditions and of the size and location of possible cracks, $\mathcal{G}_c$-type fracture strength measurements assist solution of fracture problems in real structures, for example, the crack arrest problem in pressurized cabin jet aircraft, sudden fracture of pressure vessels, and improvement in the balance between yield stress and fracture toughness.

Acknowledgments:

The authors are indebted to Mr. Frank W. Bird and Mr. E. O. Cook, U. S. Naval Research Laboratory, for their skillful technical work in performing the bulk of the experimental work reported here. Discussions of fracture strength measurements with Mr. Boris Wundt and others of the General Electric Co., Large Steam Turbine-Generator Department, were most helpful in clarifying the influence of specimen thickness.

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DISCUSSION

Mr. F. A. McClintock.[1]—It is interesting to note that an estimate can be obtained of the size of the plastic zone at which the crack becomes unstable. One might expect that the actual radius of the plastic zone, R_p, would be overestimated by Eq 9, based on the Neuber hypothesis, and underestimated by Eq 14, especially in view of the results obtained in shear.[2] Taking R_p to be defined as:

$$R_p = \frac{\mathcal{K}^2}{\sigma_y^2},$$

and evaluating $\mathcal{K}^2$ from Eq 15, yields:

$$R_p = \frac{E\mathcal{G}_{1c}}{(1 - \nu^2)\sigma_y^2}.$$

It should be noted in this equation that the yield strength of the material, σ_y, may be considerably raised by elastic constraint.[3] For the three materials shown in Fig. 6, assuming yield strengths of 72,000, 46,000, and 88,000 psi, respectively, and taking an elastic constraint factor of 2.5, the above equation yields radii of the plastic zone of 0.04, 0.26, and 0.08 in., respectively, for the limiting case of very thick plates where plane strain exists. For the case where the plate is thinner, the radius of plastic zone will be much larger, not only due to the larger values of $\mathcal{G}_c$, but also due to the absence of elastic constraint. Thus there is a plastic zone present around the tip of a crack, small compared to the size of the part, but large compared to the grain size of the material. The fact that the plastic zone is small compared to the size of the part means that the elastic stress field at a moderate distance from the plastic zone is still well described by Eqs 1 to 3 of the paper. The fact that the plastic zone is large compared to the grain size means that the microscopic theory of plasticity should be of help to us in determining what is happening even closer to the tip of the crack, and thus explaining why it is that cracks initially stable become unstable as the stress level is increased.

Could the authors comment on the reason for the decrease in the $\mathcal{G}_c$ value with decreasing thickness, shown in Fig. 9?

It should be pointed out that in the case of thin sheets, although the stress distribution may be one of plane stress in most regions, plane stress is not likely near the tip of the crack. The material immediately in front of the crack is constrained from thinning down by the relatively low-stressed material along the sides of the crack. This results in shear stresses which are neglected in the case of plane stress. For these stresses to be negligible, the thickness of the material would have to be smaller than the radius of curvature at the tip of the crack, which would be very small indeed.

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[1] Associate Professor of Mechanical Engineering, Massachusetts Institute of Technology, Cambridge, Mass.

[2] J. A. H. Hult and F. A. McClintock, "Elastic-Plastic Stress and Strain Distributions Around Sharp Notches Under Repeated Shear," IXth International Congress for Applied Mechanics, Vol. 8, pp. 51–58 (1957).

[3] G. R. Irwin, "Analysis of Stresses and Strains Near the End of a Crack Traversing a Plate," *Journal Applied Mechanics*, Vol. 25, p. 299 (1958), Discussion.

CHAIRMAN R. E. PETERSON.[4]—One can also look at this as a matter of critical crack length. I understand that the ship failures were mostly at low temperature, but I believe there were also some at essentially room temperature. I seem to recall, looking at the authors' interesting work, that the critical crack lengths for ship plate would be quite long, not $\frac{1}{4}$ in. or the length of some small weld defect, but rather long,—several feet, even at reduced temperature. I believe I have also seen some consideration where the hatch was considered as part of the opening, with the cracks extending at the hatch corners. I wonder if the authors would comment on the critical crack length in ship construction.

MR. C. MYLONAS.[5]—We have often had the opportunity of discussing this problem with the authors, and I do not think we came to a conclusion. The work presented by the authors is based on Mr. Irwin's energy theory of fracture, which is one of the few rational analytic approaches to the complex problem of brittle fracture. It is hence of extreme interest, and I always hope that it will be brought to give an answer also to the problem of brittle fracture initiation—a fundamental aspect of fracture.

During initiation, the theory presented says, in effect, that if there is more available energy than required for the fracture process, including plastic deformation, then the crack will run. This is a balance of equilibrium governing fracture. There are other examples where this condition is not sufficient. There is the very simple example of two water reservoirs of different heights, joined by a syphon. There is more energy in the difference of water levels than the friction in the tube, but the water does not run unless some more energy is supplied to make the water rise to the highest point of the syphon. So balance or excess of available energy, although a necessary condition, may not be sufficient for initiation of a fracture.

I am in full agreement with this theory after the crack starts running. Then it should be extremely useful. But in this process of starting a fracture from static conditions I do not see how only an energy consideration could be sufficient. It is certainly a necessary consideration, but is it a sufficient one?

The other point is that, if we come to the conclusion that $\mathcal{G}$ can be usefully applied, and it appears to be so, its magnitude would depend also on other factors, and one of these is prestraining. I should mention briefly that we have prestrained in compression plates already notched and then pulled them at a temperature below transition range, and got fractures at average net stress level of 37 per cent of virgin yield. We also got some arrested cracks at something like 10 per cent of virgin yield strength, cracks varying from $\frac{1}{4}$ to 3 in. in length. Such cracks occurred at 10 per cent of virgin yield strength, stopped, and the whole plate did not fracture until full yield strength occurred across the remaining section. So we had a deeper crack not fracturing at a higher stress, whereas a shorter crack (the initial notch) cracked at an average net stress 10 per cent of virgin yield strength. It is obvious that $\mathcal{G}$ is not a constant for the material.

MR. G. R. IRWIN (*authors' closure*).—The discussion by Mr. McClintock provides additional clarity to the relationship of plastic strain to governing stress conditions near the leading edge of a crack. The analysis of these plastic strains with the aid of macroscopic plasticity theory would be helpful indeed in permitting a more detailed description of the relatively abrupt increase in speed of crack extension with increase of the crack-extension force. With regard to the decrease of $\mathcal{G}_c$ with decrease of specimen thickness shown in Fig. 9, the text of the paper directs attention to "a tendency of the plastic strain zone to extend away from the crack line by an amount proportional to the plate thickness." In this extreme of the plane stress mode of fracturing, crack extension occurs through regions of the sheet which have undergone substantial and, presumably, critical amounts of local thickness reduction. For a fixed critical plastic strain leading the crack extension process one would expect the extent of the local thickness-reduction region to be proportional to the sheet thickness. The fixed critical plastic strain assumption is a gross over-simplification. Nevertheless, for α values small compared with unity, it permits one to predict a decrease of $\mathcal{G}_c$ with plate thickness as was observed.

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[5] Associate Professor of Engineering, Brown University, Providence, R. I.

In response to the discussion by Mr. Peterson, if one assumes the gross average tensile stress across the ship steel plate containing the starting crack is in the range from 15,000 psi to 35,000 psi and that a plane strain $\mathcal{G}_c$ value of 100 lb per in. applies, then, from Eq 24 of the paper, the expected half length of the starting crack is in the range from 4.2 in. to 0.8 in. The starting crack size for a shear fracture mode may be about 40 times larger corresponding to a $\mathcal{G}_c$ value of about 4000 lb per in. The corresponding half length of the starting crack for propagation as a shear fracture then would lie in the range from 14 ft to 2.5 ft. Although the hatch opening size unquestionably adds to the effective length of a crack extending from one of its corners, the conditions for crack propagation can be readily met without this assistance. A crack initiating in a zone of damage and residual stress associated with welding is running as it emerges from the residual stress zone. Because the straining is rapid, dynamic elevation of the yield stress occurs, the value of $\mathcal{G}_{Ic}$ may be somewhat reduced and the α value may be large enough for continued motion of the crack in a plane-strain mode of fracture. Under these conditions the critical half-length beyond which emergence from the influence of the residual stress field cannot arrest the crack is on the order of a few inches.

Evidently Mr. Mylonas considers that an analysis which assumes a crack to be already present is of little value in explaining the initiation of this crack. This viewpoint is not uncommon. However, large cracks form from the spreading and joining of crack-like openings at much smaller scale. The progressive extension of a crack is accomplished through the spreading and joining of small openings in advance of the leading edge. Thus, although their applicability may be limited, the fracture mechanics analysis procedures discussed in the paper are nevertheless of some value in discussions of crack initiation.

It is believed Professor Mylonas has based his discussion primarily upon the modified Griffith-theory idea suggested in references (8) and (12) of the paper. The present paper is essentially descriptive and does not depend upon a "fracture theory." The central idea is this. Given the problem of estimating the strength of a component containing a crack, one can consider a direct experimental approach to finding the answer. One can produce a crack in a specimen of the component material and make a laboratory measurement of its strength. However, the stress distribution in the laboratory specimen in general differs from that in the structural component. It is therefore desirable to make the analysis of the laboratory test in a particular way which will permit translation of the results in terms of loads on the structure. The concepts represented by the symbols $\mathcal{K}$ and $\mathcal{G}$ were introduced and employed primarily because they provide a convenient and appropriate language both for expressing the results of laboratory tests and for discussion of the conditions for crack propagation in a structure.

SIZE EFFECTS IN SLOW NOTCH-BEND TESTS OF A NICKEL-MOLYBDENUM-VANADIUM STEEL*

BY J. D. LUBAHN[1] AND S. YUKAWA[2]

SYNOPSIS

Slow notch-bend tests were performed on specimens of various sizes and notch radii cut from a large nickel-molybdenum-vanadium steel forging.

High strength values were obtained for either a small specimen with a sharp notch or for a large specimen with a mild notch, but the combination of large specimen size and sharp notch resulted in nominal strengths as low as 40,000 psi. The notch-root strain at fracture decreased with increasing size of geometrically-similar specimens. A static crack appears to cause no more weakening than a sharp machined notch.

The tests also serve to show the effects of notch depth, testing temperature, hydrogen content, and proximity of welds.

These tests were conducted as part of an investigation of the bursting of a large generator rotor and have been mentioned briefly in a previous publication (1)[3] dealing with the failure of that rotor. All the standard laboratory tests indicated that the metal of this rotor was sufficiently ductile that no significant loss of strength would be expected even in the presence of notches. Table I shows the standard mechanical properties of the steel which do suggest, however, a certain tendency towards brittleness: occasional very low reduction of area, low Charpy values, and an average notch-strength ratio of 140,000 to 108,000 = 1.30, which is distinctly less than the ideal value of 1.50 for notch-ductile materials (2). On the other hand, Table I gives no hint of weakness; although the notch tensile strength is less than ideal, it is still greater than the tensile strength.

The first test showing weakness was a bend test on a rectangular bar $2\frac{27}{32}$ in. deep by $3\frac{13}{32}$ in. wide and containing a circular crack about $1\frac{1}{2}$ in. in diameter oriented transverse to the length of the specimen and just breaking through to the tension surface. The fracture surface (Fig. 1) shows clearly the geometry of the crack, since the crack surfaces were heat tinted in welding specimen extensions to increase the span. The nominal bending strength[4] of this specimen was

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* Presented at the Sixty-first Annual Meeting of the Society, June 22–27, 1958.

[1] General Electric Co., Knolls Atomic Power Laboratory, Schenectady, N. Y.

[2] General Electric Co., Materials and Processes Laboratory, Schenectady, N. Y.

[3] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 676.

[4] Nominal stress is calculated assuming elastic behavior and ignoring stress raisers.

90,000 psi. This is considerably less than the notch tensile strength of 140,000 psi and somewhat less than the tensile strength. Since the weakness might have been due either to the sharper "notch" or to the larger size, compared with the notch tension test specimens, it was decided to test notch-bend specimens having various combinations of notch radius and size. Sharp machined notches were considered to be sufficiently severe stress raisers, since other data (3) had indicated that heat treating cracks are no more severe than sharp notches. Although earlier results (4) regarding size effects on the strength of mild steel appeared later (5) to be associated with brittle crack propagation behavior rather than brittleness, it was assumed in this investigation that size was one of the most important factors, and consequently the broad size range from 0.2 to 9 in. was investigated. Bending was used as a convenient means of obtaining high nominal stresses with a limited testing machine capacity.

TABLE I.—ROOM-TEMPERATURE MECHANICAL PROPERTIES OF THE ROTOR STEEL.

(0.30 per cent Carbon, 2.53 per cent Nickel, 0.55 per cent Molybdenum, 0.07 per cent Vanadium)

	Near the Bore				Near the Surface
	Radial		Tangential	Longitudinal	Radial
UNNOTCHED TENSILE PROPERTIES					
0.02 per cent Yield Strength, 1000 psi	86	84	81	82	82
	82	82	83	86	82
	82	80	83	...	...
	...	...	82	...	...
Tensile Strength, 1000 psi	110	110	108	111	108
	105	108	108	112	108
	110	108	91	...	...
	108 average		109	...	...
Per Cent Reduction of Area	6	15	14	27	35
	4	14	10	26	46
	18	23	2	...	...
	...	...	18	...	...
NOTCHED TENSILE STRENGTH (0.714 IN. OUTSIDE DIAMETER 60 DEG, 50 PER CENT, 10-MIL ROOT RADIUS)					
	143		132	142	...
	136		139	136	...
	140 average				
CHARPY V-NOTCH IMPACT ENERGY, FT-LB					
	4		6	6	...
	5		6	10	...

EXPERIMENTAL PROCEDURE

Material:

The composition of the forging and its standard mechanical properties are given in Table I. The fabrication and heat treatment of the forging (described in Reference (1)) resulted in a tempered bainitic microstructure. The ductility and impact values in the radial or tangential direction at the bore are somewhat lower and less consistent than values near the surface or in the longitudinal direction, but the other properties indicate that the forging is quite homogeneous and isotropic. The Charpy V-notch transition temperature is considerably above room temperature: about 150 C (302 F) for 50 per cent fibrous fracture and 85 C (190 F) for half maximum energy.

Specimens:

The specimens were radial and the long direction of the notch was tangential with respect to the forging axis. The notch was usually near mid-radius of the forging. The notches had a 45-deg included angle with depths ranging from 2 to 50 per cent of the gross beam depth and root radii ranging from 0.225 in. to less than 0.001 in. Careful machining and inspection procedures insured the proper notch radii. Extensions were welded onto some specimens, and in a few such tests the strength was less than in unwelded specimens.

Unnotched specimens of several sizes were also tested, including small specimens of considerable width ($\frac{3}{16}$ by $1\frac{1}{2}$ in.).

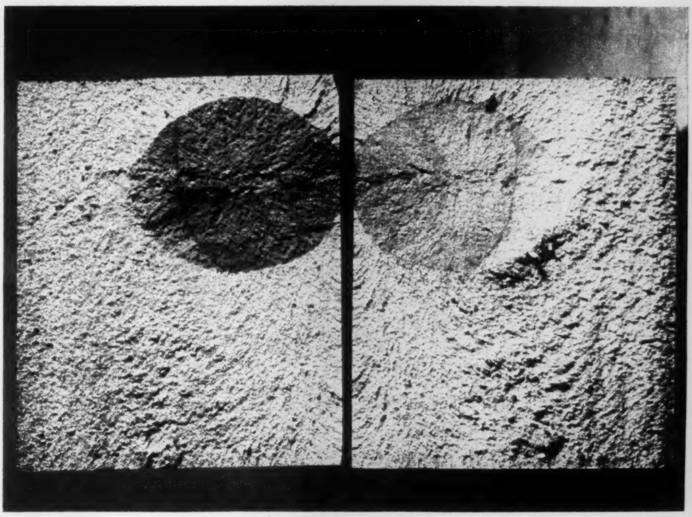

Fig. 1.—Fracture Surface of a Large Cracked Bend Specimen.

Testing:

The specimens were supported as a simple beam and loaded opposite the notch (three-point loading) at the slow rates characteristic of hydraulic testing machines. Three of the 9-in. specimens were tested in a 2000-ton hydraulic press, which was calibrated at the time by using SR-4 gages on an unnotched beam. In a previous test of this beam up to 300,000 lb on a tension testing machine, the load calculated from these strain gage readings checked the testing machine load within 2 per cent, indicating a reliable calibration. However, two later calibrations of the press by a different method gave results 17 per cent higher than the earlier one, suggesting some question of the earlier calibration.

All tests were at room temperature, except for two small specimens tested at −30 C.

On specimens geometrically similar to a standard V-notch Charpy specimen, the plastic bend angle at fracture was determined by either an intermittent or a continuous method. Plastic bend angle was converted to notch ductility, using a calibration (6) which depends on the law of similitude.

In the intermittent method, plastic bend angle was measured directly after unloading from various loads, and the curve of plastic bend angle *versus* load was extrapolated to the failure load (see also Reference (3)).

In the continuous method, the motion of the loading knife edge relative to the supports ("deflection") was measured continuously as a function of load. This motion consists mainly of specimen deflection but includes elastic distortions in the loading and support members and specimen yielding at the contact points. The elastic distortion is presumably proportional to load, and the yielding effect was found to level off eventually.

Figure 2 shows typical continuous measurement method nominal stress-bend angle curves obtained by converting the load to nominal stress using $S = Mc/I$ and the "deflection" to bend angle (equal to twice the angle whose tangent is the ratio of deflection to half the span). After the initial non-linearity due to the transient effects from the knife edges digging in, the curve is linear for a while, presumably representing elastic distortion of the specimen and knife edges. Deviations from linearity were assumed to be the "plastic bend angle" which would have been observed on unloading, and which can be converted to plastic notch strain as in the interrupted tests. At large plastic strains, the two methods of measuring plastic notch-strain yield essentially the same results (Fig. 3). The values at small plastic strain by the continuous method

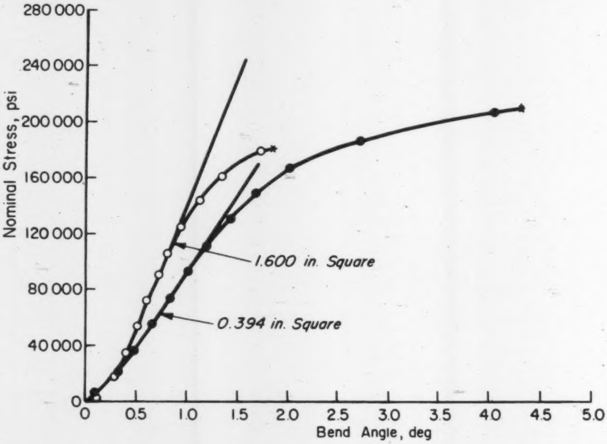

FIG. 2.—Nominal Stress *versus* Bend Angle Curves Derived from Load-Deflection Data Taken During Continuous Three-Point Loading of Nickel-Molybdenum-Vanadium Steel Specimens That Are Geometrically Similar to a Standard Charpy-Bar. Asterisks denote fracture points.

are doubtful, because of the uncertainties in fairing the points near the linear region. The intermittent method is more reliable in this range since the plastic bend angle is measured directly.

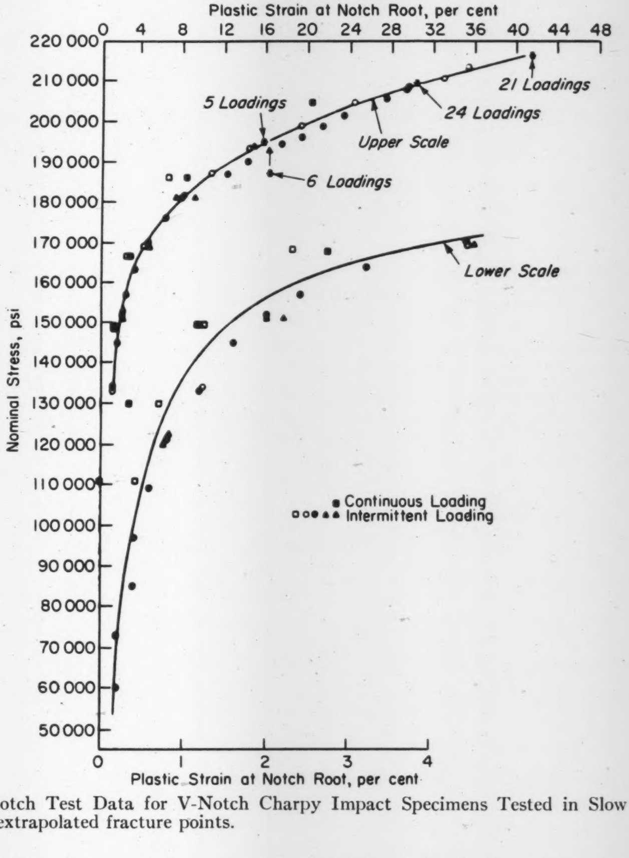

FIG. 3.—Notch Test Data for V-Notch Charpy Impact Specimens Tested in Slow Bending. Asterisks are extrapolated fracture points.

TEST RESULTS

Size Effects in Geometrically Similar Specimens:

Figure 3 shows nominal stress *versus* notch strain curves for standard V-notch Charpy specimens. Although the curves virtually coincide, the fracture point depends considerably on the number of loadings. Apparently the ductility (strain at fracture) can be improved by loading to successively higher loads below the fracture load, as with "coaxing" in fatigue behavior (7). One specimen failed on reloading at a lower load than had been reached previously.

Note in Fig. 3 that the plastic notch strain at a nominal stress of 60,000 psi is less than 0.2 per cent. This is difficult to understand, considering Brown's photoelastic data (8), which show that the local elastic stress is five times the nominal stress. This means that plastic

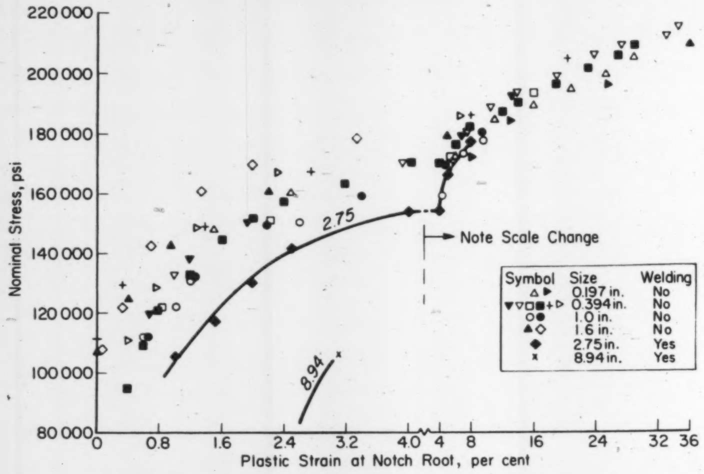

FIG. 4.—Deformation Behavior of Slow Notch-Bend Specimens of Various Sizes Geometrically Similar to V-Notch Charpy Specimen.

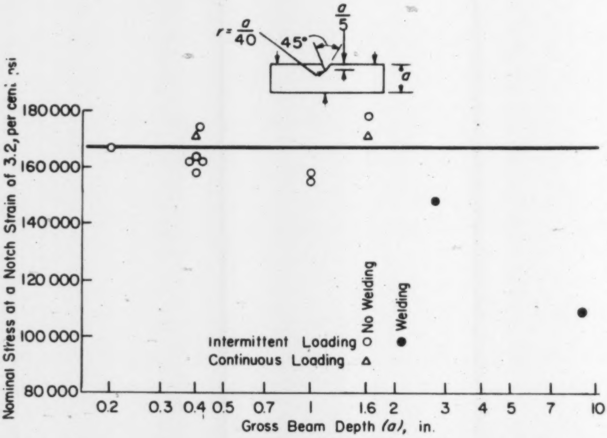

FIG. 5.—Effect of Size on the Deformation Resistance of Notch-Bend Specimens That Are Geometrically Similar to a Standard V-Notch Charpy Impact Specimen.

flow should begin at a nominal stress of 85,000/5 = 17,000 psi for a yield strength of 85,000 psi, and one would expect considerably more flow than 0.2 per cent by the time the nominal stress reached 60,000 psi.

Figure 4 shows nominal stress *versus* notch strain curves for specimens that are geometrically similar to the standard Charpy-bar. Within the limit of the scattering, the unwelded specimens define a single curve, while the welded specimens tend to follow a lower curve. The situation is shown more clearly in Fig. 5, which is a crossplot of Fig. 4 at 3.2 per cent plastic strain. Figure 5 shows that there is no effect of size on the deformation resistance, thereby confirming the law of similitude, but that welding may cause the nominal stress for a given notch strain to be abnormally low. Although the early part of the nominal stress curve for the 2.75-in. welded specimen is low, the fracture point does lie on the average curve for unwelded specimens, and consequently the fracture strength does not need to be corrected for the welding effects. For the 8.94-in. specimen, however, the fracture strength has to be corrected from 105,000 psi to about 165,000 psi, because of welds in close proximity to the notch. For this large specimen, strain measurements at the notch root before and after welding revealed that residual stresses were responsible for the effect of welding on the notch bend strength. Scribed lines 0.1 in. apart tangentially were found to be about 1 per cent farther apart after welding (average of 0.94, 1.41, and 0.64 per cent for three different sets of lines), thus indicating the introduction of very high residual stresses by welding.

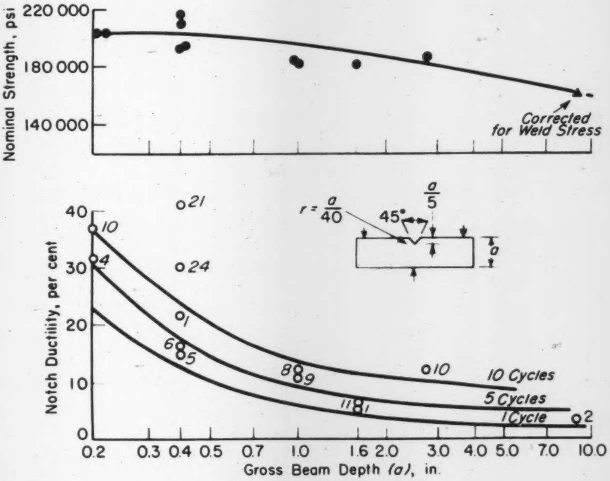

Fig. 6.—Fracture Data for Slow Notch-Bend Specimens That Are Geometrically Similar to V-Notch Charpy Impact Specimens. Numbers adjacent to points are number of sucessively higher loading and complete unloading cycles prior to fracture.

Figure 6 summarizes the fracturing data for specimens of various sizes geometrically similar to a standard Charpy bar. The notch ductility values in the lower graph have been fitted by a family of curves corresponding to different loading histories. Only the curve for 10 loading-unloading cycles is defined by enough points to show the trend with size and the other curves were drawn roughly parallel to this one. Ductility decreases tremendously with size and appears to approach zero asymptotically. The values for the smallest notched specimens are almost as high as for small, wide unnotched specimens (32 and 34 per cent for two tests). For two loadings of a 9-in. specimen, the ductility was estimated at only about 3 per cent from extrapolation of SR-4 gage data, as shown in Fig. 7.

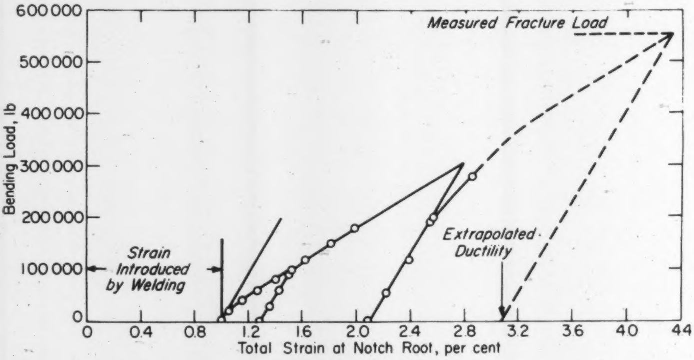

FIG. 7.—Notch-Bend Test Data for 8.94-in. Square Beam with Notch Sharpness = 1, Span: 57 in.

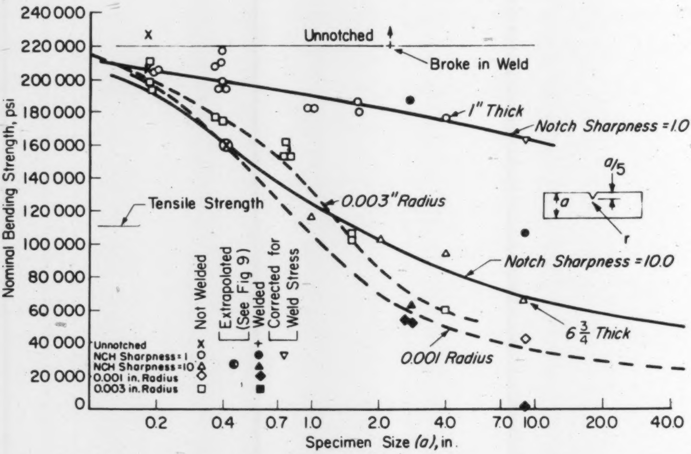

FIG. 8.—Slow Notch-Bend Strength *versus* Size and Notch Sharpness for 20 per cent Notch Depth.

The effect of loading history on notch-bend strength is small (upper graph of Fig. 6), and so only one trend curve has been drawn. There is a 20 per cent loss of strength over the size range from 0.2 to 9.0 in. The smallest notched specimens have a nominal strength equal to that of small unnotched specimens (205,000 and 227,000 psi for two tests). In Fig. 6 there is but little loss of strength corresponding to a very large effect on ductility, because of the comparative flatness of the curves in Fig. 4 beyond about 3 per cent. If the ductility fell below about 1 per cent, the effects on strength would be much greater.

Figure 8 shows the notch bend strength

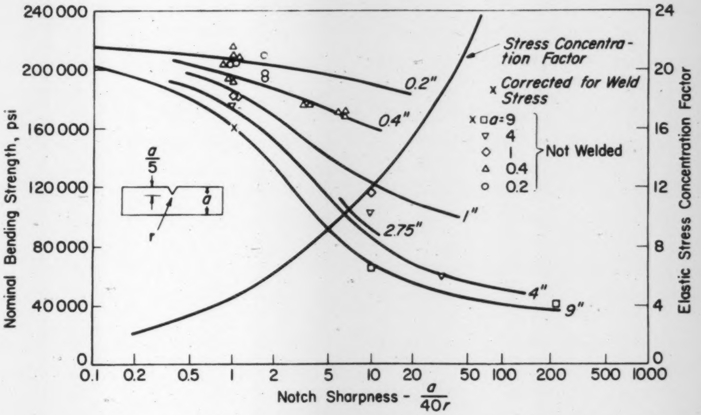

FIG. 9.—Effect of Notch Sharpness on Bend Strength of Specimens with 20 per cent Notch Depth

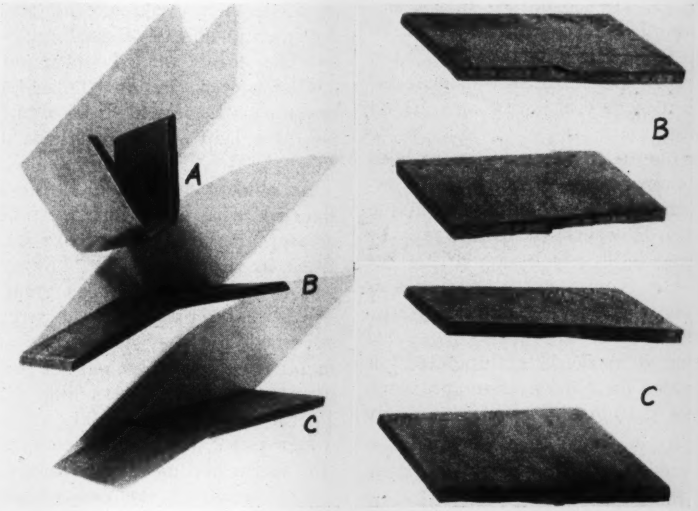

FIG. 10.—$\frac{1}{8}$ by 2 in. Bend Tests of 0.36 per cent Carbon, 1.6 per cent Manganese, 2.2 per cent Chromium, 3.6 per cent Nickel Steel Austenitized at 1725 F, Oil Quenched, Tempered at 1220 F, and Temper Embrittled 16 hr at 878 F, Rockwell Hardness C 30. Specimen A Had No Crack and a Nominal Bend Strength at About 1 per cent Strain of 198,000 psi. Specimen B had a Short Diagonal Crack and a Nominal Bend Strength of 212,000 psi. Specimen C had a Long Transverse Crack and a Nominal Bend Strength of 212,000 psi.

of all specimens with 20 per cent notch depth. There are three series of tests with specimens geometrically similar to each other but with three different values of notch sharpness (the solid curves). Notch sharpness is defined as $a/40r$, where r is root radius and a is gross beam depth. With this definition, specimens geometrically similar to the standard Charpy bar have a notch sharpness of unity, specimens with ten times smaller root radii have a notch sharpness of 10, and unnotched specimens have a notch sharpness of zero. Test results for a notch sharpness of unity from Fig. 6 are shown in the upper part of Fig. 8 again for comparison purposes. There is little, if any, effect of size on strength for unnotched specimens, a moderate effect for specimens with unit notch sharpness, and a very large effect for a notch sharpness of 10. Apparently the unnotched bending strength is approached as an asymptote as the size of the notched specimen diminishes, and it might be envisioned as an "ideal" bend strength. As the size increases from $\frac{3}{16}$ in. to 9 in., the strength falls 27 per cent and 70 per cent below ideal for notch sharpnesses of unity and 10 respectively.

The strength of small specimens with a notch sharpness of 10 cannot be determined directly because of machining difficulties. However, the values may be determined indirectly by replotting the data of Fig. 8 with notch sharpness as the abscissa as in Fig. 9, and extrapolating to sharper notches. The circled X in Fig. 8 marked "extrapolated" is such a value for a 0.4 in. size and notch sharpness of 10 in Fig. 9; it is accurately located by a short extrapolation of a well-defined curve. The accurate definition of the curves in Fig. 9 is aided by the fact that they must all approach the unnotched strength as an upper limit.

Figure 8 shows that small specimens with sharp notches and large specimens with mild notches are strong, but large specimens with sharp notches are weak. The effects of exceedingly sharp notches will now be discussed.

Size Effects in Specimens with Very Sharp Notches:

The dashed curves in Fig. 8 represent two series of tests each with constant notch radius. These curves are considerably steeper than those for geometrically similar specimens, because of the combined influences of increasing notch sharpness ($a/40r$) and size. For the worst combination of size and notch sharpness ($a = 9$, $r = 0.001$) the notch bend strength is only 43,000 psi, or only one fifth of that for small specimens.

The curves in Fig. 8 are defined by points from unwelded specimens. The strengths of welded specimens were sometimes near and sometimes much below the curves, probably depending on the proximity of the welds to the notch. A particularly striking example of the detrimental effects of welding is shown by the 9-in. welded specimen with 0.001-in. root radius. This specimen broke with a nominal stress of only 1000 psi, or a load only three times its own weight!

As evident from Fig. 9, the loss of strength with notch sharpness becomes greater as the specimen size increases. Even in the largest size, however, the loss of strength is not as great as in brittle materials, where the strength is governed by the stress concentration factor. In the steepest part of the curve, over the notch sharpness range from 1 to 10, the strength loss is only a little over a factor of 2, while the stress concentration factor becomes three times as large.

The shape of the curves in Fig. 9 indicates very little additional weakening from notches sharper than the sharpest one tested. This conclusion is partially supported by the previously mentioned result for a cracked specimen, Fig. 1, where the radius at the edge of the crack is presumably exceedingly small. This specimen was somewhat stronger, if anything, than a specimen with a sharp machined notch (85,000 psi as compared with an interpolated value in Fig. 8 of about 70,000 psi for 0.001-in. absolute notch radius). The greater strength may be due to the circular geometry of this crack as compared with straight notches.

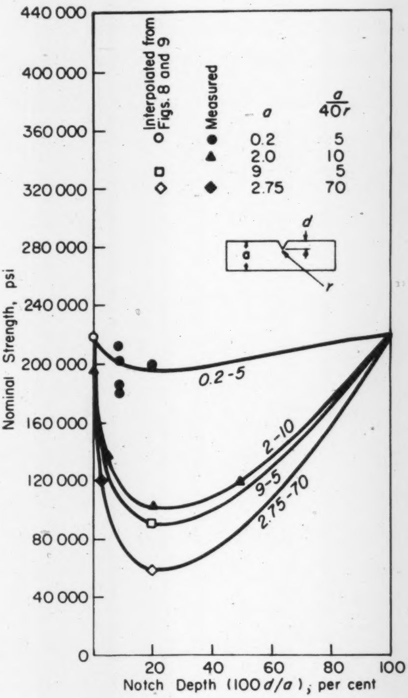

FIG. 11.—Effect of Notch Depth on Nominal Bend Strength. Numbers Adjacent to Curves Indicate Gross Beam Depth, *a*, and Notch Sharpness, *a*/40*r*.

The fact that specimens with stationary cracks are often no weaker than sharply notched ones has been observed previously (3) for some spin tests. Also, it had been found earlier that a transverse crack on the tension side of a temper embrittled bend specimen did not reduce the bending strength, in spite of the fact that the cracked specimens exhibited extreme brittleness (Fig. 10).

Notch Depth Effects:

Figure 11 shows some results for notch depths other than 20 per cent. For 2-in. bars with a notch sharpness of 10, the strength falls rapidly with notch depth, reaches a minimum at 20 to 30 per cent and increases again at 50 per cent. Notch tension tests of other high-strength steels also show a similar minimum (9), though at a larger notch depth. The increase in strength beyond the minimum may be due either to the notch sharpness effect or to the size effect. For a constant gross beam depth, *a*, and a constant root radius, *r*, an increase in notch depth, *d*, will result both in a decrease in size (defined in this case as net depth, or *a-d*) and a decrease in notch sharpness (defined in this case in relation to the net depth $\frac{a-d}{r}$). Both factors would contribute to increased strength but apparently the 16 per cent increase beyond the minimum in Fig. 11 is primarily due to the size effect. Figure 8 shows that a 12 per cent strengthening would be expected for a size reduction corresponding to an increase in notch depth from 20 to 50 per cent. For 100 per cent notch depth, the strength would be expected to rise to a high value again from either size or notch sharpness effects.

Effects of Hydrogen:

Since small specimens lose hydrogen rapidly, at the time of testing the large specimens could conceivably contain substantially the same amount of hydrogen as the original forging, while the small ones were essentially hydrogen-free. If this steel were extremely susceptible to hydrogen embrittlement, the size effect could be attributed to differences in hydrogen contents.

To investigate this possibility, several small 0.192-in. square specimens with 9 per cent notch depth and notch sharpness of 5 were cathodically charged with hydrogen at 5 amp in 4 per cent H_2SO_4 + As solution. After charging, they were stored in liquid nitrogen until testing. Slow bend tests were completed within 1 min after heating to room-temperature in a water bath. The test results are shown in Table II.

TABLE II.—EFFECTS OF HYDROGEN.

Charging Time, hr	Estimated Hydrogen Content, ppm (10)	Nominal Bend Strength, psi	
0.	0	180 000	188 000 average
0.	0	195 000	
1.	5	158 000	160 000 average
1.	5	163 000	
20.	10	151 000	143 000 average
20.	10	135 000	

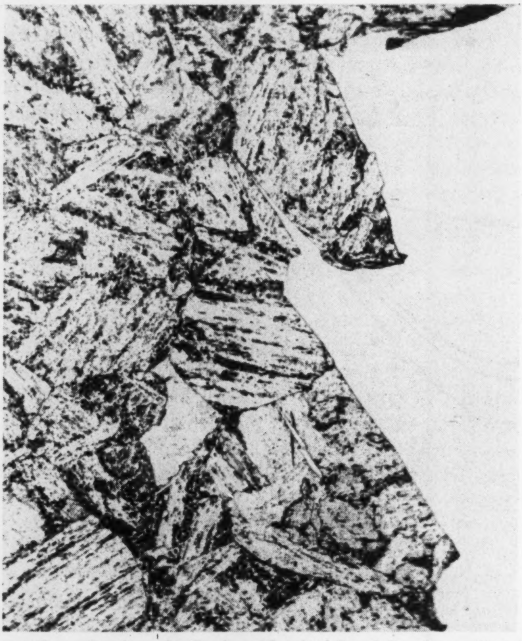

FIG. 12.—Fracture Cross-Section of a Broken Notched-Bend Specimen. Note Cleavage Mode of Fracture (× 500).

These results show that hydrogen causes a significant loss of strength; 15 per cent at 5 ppm of hydrogen and 24 per cent at 10 ppm of hydrogen. Figure 11 shows that a 9-in. bar with a 9 per cent notch depth and a notch sharpness of 5 would have a strength of only 107,000 psi, or 43 per cent less than a small, uncharged specimen. This large specimen would have only 3 ppm of hydrogen at most (the hydrogen content of the rotor) (1). Summarizing: a small specimen with 10 ppm of hydrogen is 24 per cent weaker than a small uncharged specimen, whereas a large specimen with only 3 ppm of hydrogen is 43 per cent weaker than a small uncharged specimen. From these results it must be concluded that, while hydrogen is an important factor, the size effect cannot be explained by hydrogen embrittlement alone.

Effects of Testing Temperature:

Two tests were carried out at −30 C to see whether the extremely low strengths of large specimens would be obtained in small specimens by lowering the testing temperature. The results show that specimens 0.192 in. square with a 9 per cent notch depth and a notch sharpness of 5 have strengths of 200,000 psi at room temperature and 165,000 psi at −30 C (average of 164,000 and 167,000 psi). Thus, lowering the temperature by 50 C causes a loss of strength of 17 per cent in small specimens. By contrast, Fig. 11 shows that a size increase from 0.192 in. to 9 in. decreases the room temperature strength 43 per cent.

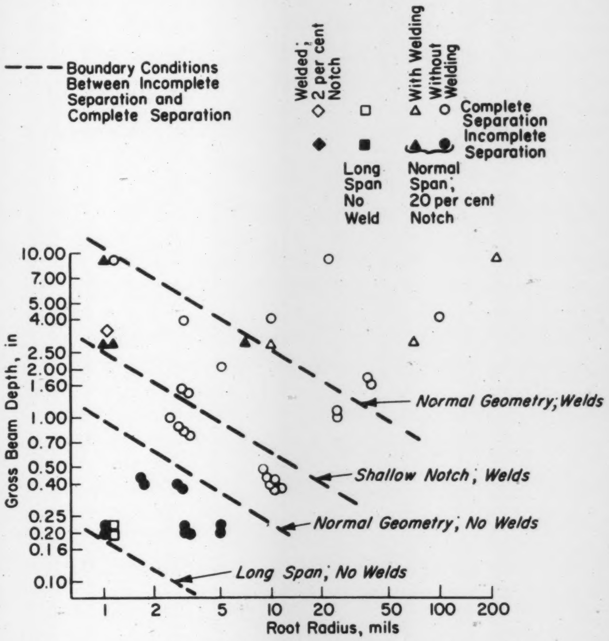

FIG. 13.—Crack Propagation Characteristics of Nickel-Molybdenum-Vanadium Steel in Notch Bend Tests.

The notch ductility at −30 C cannot be measured on these specimens with notch sharpness different from unity but the measured bend angle corresponded to 5 per cent strain for unit notch radius and presumably more for the sharper notch while the ductility of large bars is only a few per cent, even for a notch sharpness of unity. Thus, lowering the test temperature by 50 C causes much less weakening and embrittlement than increasing the size by a factor of 50.

Crack Propagation Behavior:

All bars larger than $\frac{3}{16}$ in. square broke suddenly, with a loud noise, and without any warning in the form of gradually-developing cracks. In the

$\frac{3}{16}$-in. bars a few short discontinuous cracks near mid-width could be found with a microscope just prior to sudden rupture. The sudden rupture, or "brittle crack propagation," was by cleavage, as shown in Fig. 12. Specimens showing some slight tearing before rupture also exhibited a microscopically narrow shear lip under the notch.

The cleavage crack did not always progress all the way through the specimen. Figure 13 shows that there is more tendency for incomplete fracture if the specimen is small, if the root radius is small, or if there are welds near the notch. A shallow notch or a long bending span promotes complete separation by cleavage. In short, complete separation is more likely if the stored energy is higher.

The above observations are similar to those for mild steel (5). For a given size of mild steel specimen, a sharper notch caused sudden fracture to start earlier, but to progress less far than for a milder notch. Increasing the size reduced the gradual tearing preceding the onset of sudden fracturing and promoted complete separation. However, the size for a given effect is much larger in mild steel. A 4-in. mild steel specimen still exhibited some gradual tearing and a shear lip, while the rotor steel specimens showed none of these features if the size was above about 0.4 in.

DISCUSSION

There exists much confusion in describing the fracturing behavior of steels due to the use of different criteria of "brittleness." One criterion is the complete absence of plastic strain prior to fracture, as with hardened tool steel. Another is low energy absorption in fracturing. Still another is the suddenness of crack propagation and the appearance of the fractured surfaces.

If one defines "brittleness" as lack of any plastic strain prior to crack initiation, then this steel is not brittle. The ductility measurements show greater or lesser plastic strain prior to fracture, depending on size and possibly notch sharpness, but always a finite plastic strain. On the other hand, if one defines "brittleness" as propensity to sudden crack propagation by cleavage, the material is practically 100 per cent brittle at room temperature over the size range investigated. This difference between "ductility brittleness" and "appearance brittleness" has been noted in notched bend tests of mild steel (11). In the case of mild steel, however, increasing the specimen size did not reduce the notch ductility as it did for this rotor steel, although the appearance brittleness did increase with size.

In this rotor steel, the notch ductility essentially equals the wide, unnotched bend ductility if the notch-bend specimen is sufficiently small, but the notch ductility becomes progressively less than the unnotched ductility as the size of the notch bend specimen increases. The fact that notch ductility equals unnotched ductility under some conditions and is much less under other conditions has been observed previously (3,11). In another alloy steel, the notched and unnotched ductilities were essentially equal for the one heat treatment and greatly different for another heat treatment (3). In mild steel (11), notched and unnotched ductilities are equal at room temperature but at lower temperatures the notch ductility is much less than the unnotched ductility. In all these cases, the fracture was observed to start at the surface when the notched and unnotched ductilities were equal, while surface fracture in notched specimens could never be observed when the notch ductility was less than the unnotched ductility. These observations have been explained (3,11) in terms of triaxiality (tension in all three principal directions), which exists below the notch root of a notch bend specimen but does not exist in unnotched bend specimens. If the metal is sensitive to triaxiality, a crack will start below the notch root, even though the strain is less there than at the surface, and in that case the surface strain (notch ductility) may be less than the unnotched ductility. If the metal is insensitive to triaxiality, it will break at the surface, where the conditions are the same in both notched specimens and wide, unnotched specimens (plane strain). When the conditions at the fracture origin are the same in notched and unnotched specimens, it is to be expected that notched and unnotched ductility values would be equal, as observed. As yet this hypothesis of subsurface fracture initiation has not been substantiated by direct observation—only by the indirect observation that surface fracture *cannot* be observed when notch ductility is less than unnotched ductility.

According to the law of similitude of elastic and plastic deformation, geometrically similar specimens loaded at corresponding points by forces which are in proportion to cross-section will have identical states of stress and strain. This means that geometrically similar specimens should have the same nominal bend strength if fracture depends only on the local *magnitude* of stress or strain. Since this is not the case, the important factor contributing to the greater weakness of large specimens may be the increased volume of critically strained metal in large specimens, analogous to suggestions by Peterson (**12**) for fatigue failure. The reason for this effect of the increased volume of critically strained metal is not clear now, but one possibility, deriving from the statistical theory of fracturing (**13**) is the increased probability of a serious "defect" occurring in the metal. Another possibility is that a slowly tearing crack can become of sufficient size to initiate brittle crack propagation without growing out of the critically strained region, if the volume of critically strained metal is sufficiently large.

CONCLUSIONS

1. The notch bend strength is high for a large specimen with a mild notch or a small specimen with a sharp notch, but as low as 40,000 psi for a large specimen with a sharp notch.

2. Beyond a certain notch sharpness, the strength does not decrease very much, and apparently a crack is no more severe than a sharp machined notch.

3. In specimens geometrically similar to the standard Charpy V-notch specimen, the notch ductility decreased from about 30 to 3 per cent over a size range from 0.2 to 9 in.

4. $\frac{3}{16}$-in. notch bend bars tear slightly at the surface before sudden rupture, and have the same ductility as wide, unnotched bars.

5. Notch ductility increased with increasing number of successively higher loading and unloading cycles prior to fracture.

6. For a given gross beam depth, the strength decreases rapidly with notch depth and reaches a minimum at 20 to 30 per cent.

7. Hydrogen-charged specimens were somewhat weaker than hydrogen-free specimens, but not nearly enough weaker to account for the observed size effect.

8. The notched bend strengths of small specimens are decreased by lowering the test temperature from +25 C to −30 C. This represents a much smaller weakening effect than a size increase from 0.2 in. to 9 in. at room temperature.

9. All but the $\frac{3}{16}$-in. specimens failed by sudden cleavage without any previous gradual tearing. The cleavage

fracture resulted in complete separation except for certain few combinations of short loading span, small size, sharp notch, deep notch, or proximity of welds.

10. There was a detrimental effect of residual welding stresses on the bend strengths of some specimens with welded-on extensions. An extreme example was a large specimen with a sharp notch which had a bend strength of only 1000 psi.

Acknowledgment:

The authors are indebted to E. L. Fogleman, F. Howenstein, D. R. Mack, and Mrs. H. L. Couch, for assistance in the experimental work.

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DISCUSSION

MR. G. R. IRWIN.[1]—There is a similarity in the work of the authors to the crack propagation studies discussed in our paper.[2] In that paper several sets of data were shown in which net section stress was proportional to the inverse square root of notch depth, or crack simulating notch depth. As to the notch root radius, there was a comment by George Sachs regarding the Kies-Irwin viewpoint to the effect that we employ effective notch-stress concentration factors so high the strength tends to be independent of notch sharpness.

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[1] Superintendent, Mechanics Division, U. S. Naval Research Laboratory, Washington, D. C.

[2] See p. 640 of this publication.

Referring to Fig. 8 of the authors' paper, the open diamonds and squares represent the smallest notch root radii. I found that a curve in which the ordinate is inversely proportional to the square root of notch depth goes exactly through all such measured points beyond those corresponding to the smallest test bars. Evidently, in this respect, there is a close similarity between the Lubahn-Yukawa and the Irwin-Kies-Smith papers.

I agree with the authors that sharp notches do fairly well represent actual cracks. Tests of the nature of those reported by the authors were quoted by Winne and Wundt[3] as the basis for some of the $\mathcal{G}_c$ values listed in their paper. For the reciprocal square root relation which fits the smallest root radius points of the authors' Fig. 8 I calculated a $\mathcal{G}_c$ value similar to those in the Winne-Wundt paper. Possibly this is not a coincidence. I would like to know whether any of the bend test $\mathcal{G}_c$ values quoted by Winne and Wundt were derived from measurements reported by the authors in this paper.

MR. CLARENCE SMITH.[4]—Were any particular pains taken to make these small notches free from stresses due to machining?

MR. S. YUKAWA (*author*).—In reply to Mr. Irwin, I agree with him that the strengths of sharply notched specimens larger than $\frac{3}{4}$ in. square can be described by a single curve which fits the inverse square root relationship with a $\mathcal{G}_c$ value of about 125 lb per in. However, the steel used in these tests is a different one from those described in the Winne-Wundt paper.

In regard to the preparation of the notch, no special effort was made to stress-relieve the notch after machining. The small radius notches were made by first blanking out the notch and then finishing up to the required radius by several light cuts. The specimens were tested as-machined without any subsequent thermal treatment. Undoubtedly, some localized stresses due to machining exist, but their effect is believed to be minor.

CHAIRMAN W. T. LANKFORD.[5]—It is certainly gratifying to see such a large range of sizes in a size-effect study. We do not often see such an enormous variation in sizes in this type of investigation.

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[3] D. H. Winne and B. M. Wundt, "Applications of the Griffith-Irwin Theory of Crack Propagation to the Bursting Behavior of Disks," *Paper 57-A-249*, Am. Soc. Mechanical Engrs., Dec., 1957.

[4] Convair, San Diego, Calif.

[5] Chief Research Engineer, Specialty Products, Applied Research Laboratory, U. S. Steel Corp., Monroeville, Pa.

CORRELATION OF TESTS USING THE CONGRUENCY PRINCIPLE*

By J. D. Lubahn[1]

Synopsis

The congruency principle states that fracture will occur at the same nominal stress in two different objects if the nominal stress gradient at the notch root is the same in both objects and if the notches are congruent in the two objects. This concept was applied to the results of notch bend tests and notched disk bursting tests. The strengths in the two types of tests could be correlated within about 8 per cent.

The congruency principle and another fracture concept, the Griffith-Irwin theory, were also compared as to accuracy of correlation and breadth of applicability.

In the last decade or two there has come into prominence the problem of "brittle" failure of structures under static loading. These structures have been made of materials which are ductile according to standard tests, but in service there were no obvious indications of plastic flow preceding or accompanying the rapid crack propagation that resulted in complete separation. It is generally presumed that plastic flow did indeed precede failure but was localized to such a degree as not to be apparent. Consequently, the word "brittle" has been widely applied to these failures. Actually it is not the brittleness itself which is of concern but rather the associated loss of load-carrying capacity. It is common practice to design a structure for static load application in such a way that anticipated service loads will be less than that required to cause excessive distortion by plastic flow. "Brittle" failure, then, is only of concern if it causes the load-carrying capacity to be less than that for which the structure was designed.

It has been found that "brittle" fracture becomes a more serious problem as the temperature is lowered and as the size of the structure increases. The term size effect usually refers to the fact that the problem of brittle fracture is more severe in large size structures. In the past few years, a number of investigations (**1–6**)[2] have been directed toward the effects of size and temperature on brittle fracture.

One theory of the size effect in brittle fracture is the Griffith-Irwin theory of a critical energy release rate, G_c. Data supporting this theory are available in papers on mild steel by Orowan (**7**), Irwin (**8**), Wells (**9, 10**), and Battelle

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* Presented at the Sixty-first Annual Meeting of the Society, June 22–27, 1958.

[1] Knolls Atomic Power Laboratory, General Electric Co., Schenectady, N. Y.

[2] The boldface numbers in parentheses refer to the list of references appended to this paper, p. 682.

Memorial Inst. (11), and on alloy steel forgings by the General Electric Co. (12). By means of this theory it now appears possible, within certain limitations, to explain the size effect and, what is more important, to relate one test to another. Any concept that is adequate to relate one test to another will be valuable in predicting service performance from laboratory test results. The extent to which limitations must be imposed on the Griffith-Irwin theory have not yet been established. In particular, the range of sizes, types of loading, temperatures, and materials that can be handled by the theory have not been determined.

TABLE I.—COMPARISON OF NOTCH BENDING AND NOTCH DISK BURSTING RESULTS IN TERMS OF THE GRIFFITH IRWIN THEORY (**12**).

$\mathcal{G}_c$ In Notch Bending, in-lb per sq in.		$\mathcal{G}_c$ In Notch Disk Bursting, in-lb per sq in.	
For 20 Per Cent Notch	For 67 Per Cent Notch	For 0.2 in. Notch	For 1.0 in. Notch
232	247	182	133
245	286	...	240
...	...	...	127
...	...	...	250
Average 238	266	182	187
Average for all notch depths 252		184	

$$\frac{\text{Bending strength for deep notch}}{\text{Bending strength for shallow notch}} = \sqrt{\frac{266}{238}} = 1.06$$

$$\frac{\text{Bending strength, all notch depths}}{\text{Disk bursting strength, all notch depths}} = \sqrt{\frac{252}{184}} = 1.17$$

The use of the Griffith-Irwin theory in correlating test results is illustrated by the notched bend test and notched disk bursting test results shown in Tables VI and VII of reference (12). These results have been reproduced below for comparison (Table I). The comparison shows that the notch depth effects can be accounted for within 6 per cent in either type of test and that the two types of tests can be correlated within 17 per cent, if the comparison is put on the basis of nominal stress[3] (nominal stress is proportional to the square root of $\mathcal{G}_c$, as discussed in reference (12)).

Another concept for relating one test to another has been suggested by Sachs as described by the author in reference (13). According to this concept, fracture will occur at the same nominal stress in two different objects if the local initial elastic stress distribution is the same in the neighborhood of the notch root, even though conditions elsewhere are considerably different in the two objects.

In this paper a modification of Sachs' concept will be applied to notch bend tests and internally-notched disk bursting tests (3, 5, 12). This concept will be referred to as the "congruency principle." According to this concept, fracture will occur at the same nominal stress in two different objects if the nominal stress gradient at the notch root is the same in both objects, if the notches are congruent in the two objects, and if the effect of the notches on the local stress distribution does not extend beyond the nearly-linear portion of the nominal stress distribution.

In applying the congruency principle to notch bend tests and notch disk tests, it is necessary to determine the size of beam that may be considered equivalent to a given disk. This beam size may be determined from the nominal stress distributions in the beam and the disk. In calculating these nominal stress distributions, the notched beam is treated as though it were an unnotched beam having the same depth as the net depth of the notched beam, and the notched disk is treated as though it were an unnotched disk of the same outside diameter as the notched disk but with a bore having the same inside radius as the radius to the notch root of the notched disk. In the case of the disk, it is necessary, for a strictly rigorous evaluation of nominal stress and nominal stress gradient, to consider the centrifugal force of the material at smaller radii than the radius to the notch root. However, for rather shallow notches the effects of this material are quite small, and will not be considered in computing nominal stress gradient, although the small effects on nominal stress level will be considered. The fact that the effects become large for large notch depth is of no concern because the entire concept can be applied only when the notch is rather shallow.

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[3] Nominal stress is the value that would be calculated from the loads if the notch were disregarded and the metal were assumed to behave elastically.

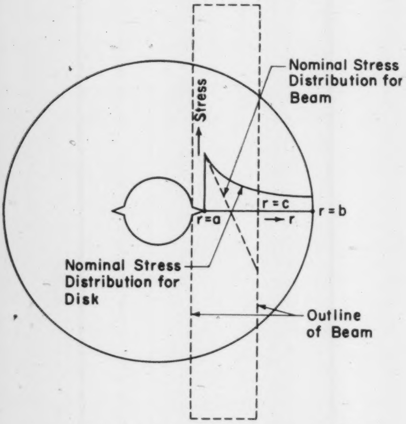
Nominal Stress Distribution for Beam
Stress
r=c
r=b
r=a
r
Nominal Stress Distribution for Disk
Outline of Beam

Fig. 1.—Schematic Diagram Showing Conditions for Equivalence of Notched Beam and Notched Disk Tests.

The tangential stress in the bored unnotched disk has the tangential stress distribution[4] indicated by the solid line in Fig. 1. This stress distribution is expressed by:

$$S_t = \frac{3+\mu}{8}(\rho\omega^2)\left(b^2 + a^2 + \frac{a^2b^2}{r^2} - \frac{1+3\mu}{3+\mu}r^2\right),$$

neglecting the effects of finite thickness. The radial rate of change is:

$$\frac{dS_t}{dr} = \frac{3+\mu}{8}(\rho\omega^2)\left(-\frac{2a^2b^2}{r^3} - 2\,\frac{1+3\mu}{3+\mu}r\right).$$

To a small crack or notch in an additional layer of metal at the inner surface of the disk, this stress distribution will appear similar to that in a similarly-notched beam in bending if the beam depth equals the distance $c - a$, such that both beam and disk have the same nominal stress gradient at $r = a$. Thus the stress gradient in the disk will be equal to that of the beam of depth, D, if:

$$D = c - a = -\left.\frac{2S_t}{\left(\dfrac{dS_t}{dr}\right)}\right]_{r=a}$$

$$D = -2\left(\frac{a^2 + 2b^2 - \dfrac{1+3\mu}{3+\mu}a^2}{-\dfrac{2b^2}{a} - 2\,\dfrac{1+3\mu}{3+\mu}a}\right)$$

$$D = 2a\left(\frac{1 + \left[\dfrac{a^2}{b^2}\right]\left[\dfrac{1-\mu}{3+\mu}\right]}{1 + \left[\dfrac{a^2}{b^2}\right]\left[\dfrac{1+3\mu}{3+\mu}\right]}\right)$$

Since in the disk tests, a/b is always a fairly small number, and the factors including Poisson's ratio are less than one, the above expression becomes practically

$$D = 2a.$$

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[4] See Timeshenko, "Strength of Materials", Vol. II, p. 247 (1941) μ = Poisson's ratio; ρ = density/acceleration due to gravity; ω = angular velocity in radians per second; r = radius; a = radius of hole; b = outside radius.

That is, the depth of the equivalent unnotched beam would be about equal to the bore diameter of the unnotched disk.

TABLE II.—COMPARISON OF NOMINAL STRENGTHS OF DISKS AND BEAMS USING CONGRUENCY PRINCIPLE.
(Data from references (**2**, **3**, **5**, and **12**))

	Forging WD1B	Forging WD	Forging WD	Forging WL
		DISK DATA		
Outside diameter, in.	24	24	9.055	19
Inside diameter, in.	3¼	3¼	1.259	3¼
Notch depth, in.	0.2	1	0.406	1
Nominal stress, psi	95 000	49 000 (WD1A, 63 F, 6 in. thick) 71 000 (WD2A, 115 F, 6 in. thick) 49 000 (WD7A, 79 F, 2 in. thick) 68 000 (WD8A, 80 F, 2 in. thick)	110 000	122 000
Average, psi		59 000		
		BEAM DATA		
Equivalent beam				
Net depth, in.	3.65	5.25	2.071	5.25
Gross depth, in.	3.85	6.25	2.477	6.25
Notch depth, per cent	5.2	16	16.5	16
Tested beam				
Net depth, in.	2	2	2	5.25
Gross depth, in.	2.5	2.5	2.5	6.56
Notch depth, per cent	20	20	20	20
Strength, psi	102 000 106 000	102 000 106 000	102 000 106 000	112 000[a]
Correction for notch depth	1.33	1.03	1.03	1.03
Correction for beam depth	0.74	0.617	0.98	1.0
Corrected strength, psi	101 000 105 000	65 000 67 000	103 000 107 000	115 000
Average, psi	103 000	66 000	105 000	
Ratio of beam to disk strength	1.08	1.12	0.96	0.93
Average error, per cent				8

[a] Extrapolated with respect to size.

In order to maintain strict congruency, the width of the beam should be chosen equal to the disk thickness, but the beam width is probably not a very critical factor.

As an example of the use of the above correlation, consider disk test WD3T from Table III of reference (12). This is 9.055 in. outside diameter, 1.259 in. inside diameter, with two 0.406 in. deep saw cuts at each side of the hole. According to the above concepts, this disk would be treated as though it were an unnotched disk of $[1.259 + (2)(0.406)] = 2.071$ in. inside diameter and 9.055 in. outside diameter. The equivalent beam would have a net depth of 2.071 in., gross beam depth of 2.477 in., and a notch depth of 0.406 in. (16.5 per cent).

The disk failed at 28,140 rpm. The corresponding nominal tangential bore stress is found by substituting $r = a = \frac{2.071}{2}$, $b = \frac{9.055}{2}$, $\rho = \frac{\gamma}{g} = \frac{0.284}{12 \times 32.2} = 0.000736$, and $\omega = \frac{28.140 \times 2\pi}{60}$ in the above equation to obtain $S_t = 108{,}300$ psi. In addition, there is a small amount of tangential stress at the bore due to the centrifugal force of the material between $r = \frac{1.259}{2} = 0.630$ in. and $r = \frac{2.071}{2} = 1.036$ in. which contains the notch; but was not considered as part of the unnotched disk in the above calculations. This exerts an internal pressure ρ_0 of:

$$\rho_0 = \frac{1}{1.036} \int_{0.630}^{1.036} \frac{\gamma}{g}\, \omega^2 r^2 \, dr = 1760 \text{ psi}$$

which causes a tangential bore stress of

$$S_t = \rho_0 \frac{b^2 + a^2}{b^2 - a^2} = \rho_0 \left(\frac{4.528^2 + 1.036^2}{4.528^2 - 1.036^2} \right) = 1960 \text{ psi}$$

The total nominal stress is $108{,}300 + 2000 = 110{,}300$ psi.

Now this strength value could be compared with that of a beam if the beam had a net depth of 2.071 in. and 16.5 per cent notch depth, that is, if data were available for the "equivalent beam." The nearest beam tests have 2 in. net depth and 20 per cent notch depth (Table VI of reference (12)), and consequently are not strictly comparable. However, the comparison can be made if small corrections for net beam depth and notch depth are made on the basis of data on a similar material. Reference (3) shows that a beam with 16.5 per cent notch depth is 3 per cent stronger than one with 20 per cent notch depth, and that a beam with 2.072 in. net beam depth (2.59 in. gross beam depth) is 2 per cent weaker than a beam with 2 in. net beam depth (2.5 in. gross beam depth). Consequently, the equivalent beam should have a strength of (1.03) (0.98) times that of the beams that were tested, or (1.03)(0.98)(102,000) = 103,000 psi and (1.03)(0.98)(106,000) = 107,000 psi. The average of these values compares favorably with the 110,000 psi nominal stress in the disk.

Table II shows the above calculations in tabular form and similar comparisons for another disk size, another notch depth, and another material. In some cases, the correction for notch depth or net beam depth is rather large, because the data are for beams considerably different from the equivalent beam. In these cases, the correlation is not quite as good, probably because the size and notch depth relationships for the material of reference (3) are not exactly applicable to other materials. In all cases, however, the correlation was at least as good as that using the Griffith-Irwin theory (Table I), and the average error in the correlation was 8 per cent. The congruency principle also has the advantage of applying to all kinds of materials, while the Griffith-Irwin theory applies only to what Winne and Wundt (12) have called "quasi-brittle materials." On the other hand, the congruency principle has one serious disadvantage: the notch geometry of the test specimen and the nominal stress gradient must exactly match that in the application. The Griffith-Irwin theory is not limited in this way.

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DISCUSSION

MR. G. R. IRWIN.[1]—The method of measuring the crack extension strength of the material in terms of the $\mathcal{G}_c$ value, as the author has noted, led to a definite numerical value which could be recorded for that particular thickness and temperature of the material. The method which the author has just described seems to be somewhat more general. From the basic nature of dimensional analysis and of the congruency principle, it would seem to include within it the approach which I was presenting and I have no complaint or disagreement with it.[2]

I should like to ask the author whether he has a suggestion as to how in his measurements and presumably on very short notches completely surrounded with plastic strain, he would place numerical values on his results?

MR. J. D. LUBHAN (*author*).—When you ask, "How can one put a numerical value on the result?" I presume that you are asking, "What material property, similar to the critical energy release rate can be determined by using the congruency principle?" The answer is that one cannot obtain a basic material property; and that is a definite disadvantage of the method. The method is purely an empirical correlation between two types of tests. Let us say one wanted to predict the bursting speed of a notched disk. What one has to do is to make a notch-bend specimen, which has the correct beam depth to give the nominal stress gradient that exists in the disk. Then one must introduce an identical notch. I did not mention this before, but the notch-bend specimen must also be the same thickness, as the disk. Finally one measures the nominal strength. Then, by assuming that the disk would have that same nominal strength as the notch-bend specimen, one could figure back to the bursting speed of the disk. In this entire analysis, no basic quantity similar to a $\mathcal{G}_c$ value, appears.

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[1] Superintendent, Mechanics Division, U. S. Naval Research Laboratory, Washington, D. C.

[2] See p. 640 of this publication.

There is one more limitation, in addition to that of requiring the correct thickness of the beam, that must be imposed upon the method: if the notch is too deep, so that the disturbance of the nominal stress distribution because of the notch, extends beyond the region where the nominal stress distribution can be considered as being a straight line, the correlation will not be valid either. Thus, the limitation of comparatively shallow notches has to be imposed on the congruency principle also.

EFFECT OF STRAIN RATE AND TEMPERATURE ON THE STRENGTH OF MAGNESIUM ALLOYS*

BY RAYMOND W. FENN, JR.[1] AND JAMES A. GUSACK[1]

SYNOPSIS

Investigation of the effect of strain rate (0.005, 0.050, 0.50 and 5.0 per min) on the strength of several rolled, extruded and cast magnesium alloys at temperatures up to 900 F reveals significant increases in strength with increasing testing speed. This study provides information for use in design of structures subjected to rapid loading. It also points out the need for standardizing the speed of testing in elevated-temperature testing.

It is known that the speed of testing of materials as well as the test temperature affects their mechanical properties. In general strength increases with increasing strain rate and decreases with increasing test temperature.

When service loads are rapidly applied, it is possible that a more efficient structure could be designed by taking advantage of the higher strengths that accompany rapid strain rates. However, many testing laboratories test material at only one, relatively slow, strain rate. Therefore, a relationship between strength properties and various strain rates could be useful to structural designers, and also to those comparing results of property determinations obtained at different strain rates from different testing laboratories. For these reasons a program was initiated to determine the relationship of the strength of certain magnesium alloys to the strain rate and test temperature.

TEST PROGRAM AND MATERIALS

Five sheet, seven extrusion, and six sand-cast magnesium alloys were tested in tension in duplicate at strain rates of approximately 0.005 per min, 0.050 per min, 0.50 per min, and 5.0 per min at test temperatures of 75 to 900 F. Chemical compositions of the alloys studied are shown in Table I. AZ31B-H24, AZ31B-0, HK31A-H24, HK31A-0, and HM21A-T8 sheet alloys (0.064 in. thick) were commercially produced. The extrusion alloys AZ31B-F, AZ61A-F, AZ80A-T5, HM31XA-F, M1A-F, ZK60A-F, and ZK60A-T5 were extruded (0.064 by 1.25 in. in cross-section) in pilot-process operations, while separately-cast test bars of AZ91C-T6, AZ92A-T6, EZ33A-T5, HK31A-T6, HZ32A-T5, and ZH62A-T5 alloys were produced by routine sand-casting procedures. The properties obtained for all alloys are within the range of properties expected for commercial material; the strength of the cast ZH62A-T5 and EZ33A-T5, however, are near the upper limits of their scatter bands while the

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* Presented at the Sixty-first Annual Meeting of the Society, June 22–27, 1958.

[1] Metallurgical Laboratory, The Dow Chemical Co., Midland, Mich.

TABLE I.—SPECTROGRAPHIC AND CHEMICAL ANALYSES OF THE MAGNESIUM ALLOYS, CHEMICAL COMPOSITION, PER CENT.

Alloy, Temper, and Form	Aluminum	Calcium	Copper	Iron	Manganese	Nickel	Lead	Silicon	Tin	Zinc	Thorium	Zirconium	Total Rare Earths	Magnesium
AZ31B-H24, sheet	3.05	<0.01	0.004	0.002	0.51	<0.0005	<0.001	<0.01	<0.01	1.03	....	....	....	Bal
AZ31B-0, sheet	3.05	<0.01	0.004	0.002	0.51	<0.0005	<0.001	<0.01	<0.01	1.03	....	....	....	Bal
HK31A-H24, sheet	<0.03	<0.01	<0.005	<0.002	0.052	<0.001	<0.001	<0.01	<0.01	<0.02	2.62	0.61	....	Bal
HK31A-0, sheet	<0.03	<0.01	<0.005	<0.002	0.052	<0.001	<0.001	<0.01	<0.01	<0.02	2.62	0.61	....	Bal
HM21A-T8, sheet	0.007	<0.01	<0.005	<0.002	0.46	<0.001	<0.001	<0.01	<0.01	<0.02	2.2	....	....	Bal
AZ31B-F, extrusion	2.85[a]	<0.01	<0.001	0.0015	0.53[a]	<0.0005	<0.001	0.010	<0.01	1.01[a]	....	....	....	Bal
AZ61A-F, extrusion	6.42[a]	<0.01	0.001	0.016	0.23[a]	<0.001	<0.001	0.013	<0.01	0.96[a]	....	....	....	Bal
AZ80A-T5, extrusion	8.56[a]	<0.01	0.001	0.020	0.26[a]	<0.001	<0.001	0.012	<0.01	0.02[a]	....	....	....	Bal
HM31XA-F, extrusion	<0.03	<0.01	0.003	0.012	1.89[a]	0.003	<0.001	<0.01	<0.01	<0.02	3.19[a]	....	....	Bal
M1A-F, extrusion	0.016	<0.01	0.003	0.009	1.39[a]	0.005	<0.001	0.014	<0.01	0.011	....	....	....	Bal
ZK60A-F, extrusion	<0.03	<0.01	0.003	0.002	0.066	0.003	0.003	<0.01	<0.01	5.59[a]	....	0.69[a]	....	Bal
ZK60A-T5, extrusion	<0.03	<0.01	0.003	0.002	0.066	0.003	0.003	<0.01	<0.01	5.59[a]	....	0.69[a]	....	Bal
AZ91C-T6, casting	8.55[a]	<0.01	0.003	0.028	0.20[a]	<0.001	<0.001	0.013	<0.01	0.76[a]	....	....	....	Bal
AZ92A-T6, casting	9.5[a]	<0.01	0.010	0.010	0.24[a]	<0.001	0.004	0.018	<0.01	2.01[a]	....	....	....	Bal
EZ33A-T5, casting	<0.03	<0.01	0.001	<0.001	0.051	<0.001	0.009	<0.01	<0.01	2.77[a]	....	0.68[a]	3.32[b]	Bal
HK31A-T6, casting	<0.03	<0.01	0.001	0.002	0.053	<0.001	<0.001	<0.01	<0.01	<0.02	3.12[a]	0.79[a]	....	Bal
HZ32A-T5, casting	<0.03	<0.01	0.002	0.001	0.055	<0.001	0.004	<0.01	<0.01	2.08[a]	2.72[a]	0.71[a]	....	Bal
ZH62A-T5, casting	<0.03	<0.01	0.001	0.001	0.057	<0.001	0.010	<0.01	<0.01	5.46[a]	1.79[a]	0.87[a]	....	Bal

[a] Denotes chemical analysis.

[b] Denotes mischmetal by chemical analysis.

TABLE II.—EFFECT OF STRAIN-RATE AND TEMPERATURE ON THE STRENGTH OF SOME MAGNESIUM SHEET ALLOYS.

Test Temperature, deg Fahr	Tensile Yield Strength, 1000 psi				Tensile Strength, 1000 psi				
	Strain per min				Strain per min				
	0.005	0.050	0.50	5.0	0.005	0.050	0.10	0.50	5.0
AZ31B-H24 Alloy									
75	30.0	30.7	31.5	32.2	40.2	40.5	40.5	40.7	41.0
200	23.2	25.1	27.1	29.1	28.5	31.2	32.1	34.1	36.9
300	14.5	17.9	20.4	22.5	14.5	20.2	21.7	24.7	28.7
400	6.5	9.7	13.9	18.6	7.3	10.2	11.4	14.8	23.0
500	3.7	6.6	9.4	12.6	4.8	6.6	7.4	9.4	14.5
600	2.0	4.2	6.5	9.3	3.2	4.5	5.1	6.5	10.0
700	1.0	2.7	4.6	7.0	2.0	3.0	3.5	4.6	7.0
800	....	1.8	3.2	4.7	....	1.8	2.1	3.2	5.0
900	....	....	2.0	2.9	....	....	....	2.2	3.0
AZ31B-O Alloy									
75	21.4	21.6	22.0	22.8	36.1	36.5	36.6	36.9	37.3
200	17.8	18.1	18.8	21.0	27.0	29.0	29.5	30.9	32.8
300	13.0	14.1	14.6	17.1	19.0	21.7	22.5	24.2	26.9
400	9.2	10.7	12.2	13.7	9.3	12.3	13.5	16.5	21.3
500	5.8	8.0	8.7	11.5	5.8	8.2	8.7	10.2	15.0
600	2.9	5.2	6.4	9.3	3.3	5.2	5.7	7.0	10.1
700	0.8	3.5	4.6	7.5	2.0	3.5	3.9	5.0	7.5
800	....	2.0	3.2	4.5	....	2.2	2.5	3.3	4.5
900	....	....	2.0	3.0	....	....	....	2.2	3.0
HK31A-H24 Alloy									
75	29.2	29.7	30.2	30.6	34.3	35.7	36.0	37.0	38.5
200	26.5	27.2	27.7	28.4	28.5	29.9	30.2	31.2	32.6
300	24.0	24.6	25.2	25.7	24.2	25.5	26.0	27.0	28.4
400	19.9	21.2	22.5	23.8	20.5	22.1	22.5	23.7	25.2
500	16.9	18.2	19.8	21.2	17.0	18.6	19.0	20.2	21.7
600	6.5	12.2	15.5	18.1	7.5	12.7	13.8	15.9	18.4
700	1.5	4.1	7.5	13.7	2.0	4.9	6.0	9.1	14.0
800	....	1.3	3.7	7.0	....	2.0	2.6	4.7	8.1
900	....	....	2.5	5.0	....	....	....	2.6	5.0
HK31A-O Alloy									
75	21.2	21.7	22.4	23.0	28.6	30.7	31.3	32.7	34.8
200	18.2	19.0	19.9	20.7	21.3	23.4	24.1	25.6	27.7
300	15.8	16.9	17.0	19.1	19.5	19.8	20.0	20.5	22.0
400	13.3	14.5	15.2	17.8	15.7	15.7	15.7	16.3	18.3
500	12.1	12.5	12.7	15.3	14.4	14.5	14.5	14.5	15.3
600	8.5	9.5	10.2	12.1	8.7	11.5	12.0	12.7	13.5
700	2.2	5.2	6.8	9.6	2.2	5.3	6.2	8.2	10.5
800	....	2.5	4.5	6.4	....	2.5	3.2	4.7	6.8
900	....	....	2.8	4.6	....	....	....	2.9	4.7
HM21A-T8 Alloy									
75	24.7	24.8	24.9	25.0	30.4	31.9	32.4	33.4	35.0
200	22.0	22.3	22.7	23.1	24.3	25.7	26.2	27.2	28.8
300	19.0	19.8	20.7	21.5	19.6	21.2	21.6	22.7	24.3
400	15.8	16.4	17.5	20.0	16.2	17.1	17.5	18.6	21.7
500	14.1	14.6	15.5	18.2	14.1	14.8	15.1	16.0	18.2
600	11.7	12.1	13.0	14.8	12.0	12.7	13.0	13.7	15.1
700	6.1	8.6	10.5	12.0	6.2	8.9	9.5	11.0	12.7
800	2.3	3.8	6.0	8.8	2.3	4.3	5.0	6.9	9.7
900	....	....	1.7	2.7	....	....	....	2.5	4.9

TABLE III.—EFFECT OF STRAIN-RATE AND TEMPERATURE ON THE STRENGTH OF SOME MAGNESIUM EXTRUSION ALLOYS.

Test Temperature, deg Fahr	Tensile Yield Strength, 1000 psi				Tensile Strength, 1000 psi				
	Strain per min				Strain per min				
	0.005	0.050	0.50	5.0	0.005	0.050	0.10	0.50	5.0
AZ31B-F ALLOY									
75	23.6	24.4	25.3	26.2	38.2	38.2	38.2	38.2	38.2
200	19.3	20.5	21.7	23.0	....	32.8	33.0	33.7	34.6
300	12.7	14.5	16.5	19.0	....	24.0	24.7	26.4	28.8
400	8.7	10.9	12.9	15.2	....	14.9	15.8	18.2	21.5
500	5.5	7.8	9.2	11.5	....	9.1	10.0	12.1	16.5
600	3.2	5.4	6.5	8.8	....	5.8	6.4	8.0	11.2
700	1.8	3.7	4.7	6.8	....	4.0	4.5	5.9	8.5
800	....	2.2	3.5	4.7	....	2.3	2.7	3.8	5.7
900	....	1.3	2.4	3.5	....	1.4	1.7	2.5	3.7
AZ61A-F ALLOY									
75	24.7	25.1	25.5	26.0	44.7	44.7	44.7	44.7	44.7
200	22.3	22.9	23.6	24.3	39.6	39.7	39.7	39.7	39.8
300	16.5	18.6	20.4	21.8	....	28.3	30.2	33.0	35.7
400	11.1	14.5	16.5	19.0	....	16.7	18.9	22.8	27.3
500	7.1	10.0	11.5	15.6	....	11.1	12.0	14.4	20.1
600	3.9	6.4	7.4	10.5	....	6.5	7.1	9.3	13.4
700	1.2	3.6	5.0	8.0	....	3.8	4.2	6.0	9.4
800	....	1.8	3.6	5.7	....	2.0	2.5	4.0	6.0
900	....	0.9	2.3	3.8	....	1.1	1.5	2.3	3.8
AZ80A-T5 ALLOY									
75	30.1	32.3	34.5	36.7	48.5	48.5	48.5	48.5	48.5
200	24.0	26.8	29.6	32.5	....	41.0	41.5	42.8	44.5
300	15.8	19.1	22.4	25.7	....	26.3	27.8	31.4	36.4
400	9.7	13.4	17.1	20.8	....	16.1	17.5	20.7	26.6
500	5.3	8.8	11.2	16.0	....	9.7	10.7	13.5	19.7
600	2.6	6.0	8.3	12.5	....	6.4	7.1	9.3	14.0
700	1.3	3.5	5.4	8.0	....	4.0	4.5	5.9	9.5
800	....	2.2	4.0	5.7	....	2.4	3.0	4.3	6.7
900	....	1.0	2.0	2.9	....	1.2	1.4	2.0	2.9
HM31XA-F ALLOY									
75	35.2	35.5	35.9	36.1	37.4	38.5	38.9	39.7	41.9
200	30.2	30.7	31.2	33.2	30.0	31.5	32.0	33.3	35.7
300	25.5	26.1	27.4	30.5	26.0	26.8	27.1	28.2	31.0
400	22.6	22.7	23.5	26.2	23.0	23.4	23.6	24.3	26.7
500	20.0	20.0	20.7	23.1	20.2	20.4	20.5	21.1	23.4
600	12.7	15.1	16.5	17.9	12.7	15.7	16.3	17.5	18.8
700	7.5	8.4	9.9	12.8	9.3	9.6	9.8	10.7	13.5
800	3.0	3.3	4.4	7.8	5.0	5.0	5.3	6.0	9.0
900	1.0	1.2	2.1	5.0	1.5	1.7	1.9	2.5	5.0

TABLE III.—*Concluded.*

Test Temperature, deg Fahr	Tensile Yield Strength, 1000 psi				Tensile Strength, 1000 psi				
	Strain per min				Strain per min				
	0.005	0.050	0.50	5.0	0.005	0.050	0.10	0.50	5.0
				M1A-F Alloy					
75	24.9	26.3	27.7	29.2	33.5	36.6	37.5	39.7	42.7
200	19.8	21.7	23.7	25.6	....	26.0	27.1	29.6	33.1
300	12.0	15.5	19.3	23.6	....	17.1	18.0	20.3	25.3
400	7.5	10.0	12.8	17.3	....	11.5	12.0	13.8	17.9
500	4.8	6.6	8.4	12.0	....	7.5	8.0	9.5	13.0
600	3.7	4.7	5.8	8.0	....	5.3	5.7	6.9	9.3
700	2.6	3.4	4.2	5.0	....	3.9	4.2	5.0	6.3
800	....	2.6	3.0	3.3	....	3.0	3.1	3.6	4.3
900	....	1.7	2.0	3.0	....	2.0	2.1	2.6	3.2
				ZK60A-T5 Alloy					
75	39.8	42.4	45.1	47.7	....	51.3	51.6	52.6	53.8
200	27.0	31.5	36.0	40.4	....	37.4	38.6	41.4	45.8
300	16.5	21.2	25.8	30.5	....	24.6	25.8	28.8	34.6
400	6.5	12.9	17.7	22.5	....	14.2	15.5	18.8	24.7
500	2.3	5.1	8.2	15.1	....	5.5	6.7	10.2	17.9
600	1.7	2.4	4.1	8.7	....	2.5	2.9	5.1	10.9
700	1.1	1.7	3.0	6.0	....	1.9	2.0	3.3	6.8
800	....	1.4	2.1	3.6	....	1.5	1.6	2.3	4.4
900	....	1.4	2.1	3.6	....	1.5	1.6	2.3	3.5
				ZK60A-F Alloy					
75	38.0	39.5	41.0	42.6	....	50.3	50.5	51.0	51.6
200	26.5	30.0	33.6	37.2	....	39.0	39.8	41.4	43.7
300	15.0	19.9	24.8	29.7	....	24.1	25.6	29.2	34.3
400	6.2	11.4	16.6	22.0	....	13.5	15.2	19.0	24.5
500	2.4	4.8	8.3	15.0	....	5.3	6.1	9.7	17.4
600	1.4	2.3	4.2	9.7	....	2.4	2.9	4.9	10.7
700	1.3	1.6	2.6	6.6	....	1.7	1.9	3.2	7.2
800	....	1.4	2.0	3.5	....	1.4	1.5	2.3	4.2
900	....	1.1	2.0	3.5	....	1.4	1.5	2.3	3.7

strength of extruded AZ80A-T5 is near the lower limit of its band.

Flat specimens with standard reduced sections[2] were made from the sheet and extrusion alloys. Round specimens having reduced sections varying from 5 in. ($\frac{3}{8}$ in. diameter) to $1\frac{1}{2}$ in. ($\frac{1}{4}$ in. diameter) long were machined from the cast alloys. The shorter specimens were necessary to obtain the higher strain rates desired at the lower temperatures.

Test Methods

A 12,000-lb capacity gear-driven testing machine (Baldwin model PTE) equipped with a Microformer stress-strain recorder (model MA1-B) and

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[2] Tentative Methods of Tension Testing of Metallic Materials (E 8 – 57 T), 1957 Supplement to Book of ASTM Standards, Part 1, p. 437.

TABLE IV.—EFFECT OF STRAIN-RATE AND TEMPERATURE ON THE STRENGTH OF SOME SAND-CAST MAGNESIUM ALLOYS.

Test Temperature, deg Fahr	Tensile Yield Strength, 1000 psi				Tensile Strength, 1000 psi				
	Strain per min				Strain per min				
	0.005	0.050	0.50	5.0	0.005	0.050	0.10	0.50	5.0
AZ91C-T6 Alloy									
75.......	19.5	19.5	19.5	19.5	....	41.0	41.0	41.0	41.0
200.......	17.3	18.7	19.1	19.2	....	38.5	38.6	38.8	39.0
300.......	14.7	15.7	16.8	17.8	....	28.1	29.3	31.9	35.6
400.......	11.9	13.1	14.5	15.8	....	18.2	19.2	21.9	26.5
500.......	9.0	10.7	12.5	14.2	....	13.0	13.8	15.9	20.2
600.......	6.5	8.2	10.1	12.0	....	9.5	10.2	12.0	15.5
700.......	3.5	5.5	7.5	9.5	....	5.6	6.3	8.2	12.0
800.......	1.5	2.6	3.0	3.0	....	2.6	2.8	3.0	3.0
900.......	....	....	....	....	....	....	....	....	....
AZ92A-T6 Alloy									
75.......	23.0	23.0	23.0	23.0	....	39.0	39.0	39.0	39.0
200.......	20.2	21.5	22.5	22.9	....	37.0	37.0	37.0	37.0
300.......	15.7	18.2	20.3	21.5	....	29.1	30.7	33.1	35.8
400.......	11.0	13.8	16.5	19.4	....	18.2	19.3	21.9	28.3
500.......	7.7	10.3	13.0	15.7	....	12.0	12.8	15.0	20.5
600.......	5.4	7.7	9.9	12.2	....	8.3	9.0	11.0	15.0
700.......	2.7	4.5	7.0	10.1	....	4.5	5.2	7.2	11.2
800.......	....	....	....	....	....	....	....	....	....
900.......	....	....	....	....	....	....	....	....	....
EZ33A-T5 Alloy									
75.......	18.0	18.0	18.0	18.0	....	27.5	27.5	27.5	27.5
200.......	14.5	15.5	15.9	16.0	....	24.0	24.0	24.0	24.0
300.......	12.2	13.7	14.4	14.6	....	23.5	23.5	23.5	23.5
400.......	10.7	11.1	12.0	13.3	....	22.3	22.3	22.3	22.3
500.......	10.0	10.1	10.5	11.4	....	19.4	19.7	20.0	20.0
600.......	9.3	9.4	9.6	10.2	....	14.4	15.7	17.3	18.0
700.......	5.8	7.0	8.2	9.3	....	7.5	8.8	11.7	14.7
800.......	2.2	3.7	5.4	6.9	....	3.7	4.1	5.6	9.8
900.......	1.0	1.4	2.1	5.0	....	1.4	1.7	2.7	6.7
HK31A-T6 Alloy									
75.......	16.1	16.3	16.4	16.7	....	33.8	34.0	34.4	35.0
200.......	15.2	15.4	15.6	15.8	....	26.6	27.1	28.2	29.9
300.......	14.4	14.6	14.8	15.0	....	25.1	25.5	26.0	27.0
400.......	13.8	13.9	14.1	14.2	....	23.5	23.5	23.5	23.5
500.......	13.0	13.1	13.2	13.4	....	20.5	20.5	20.5	20.5
600.......	12.1	12.2	12.4	12.5	....	19.0	19.0	19.0	19.0
700.......	7.1	7.6	8.1	8.5	....	12.3	12.9	14.0	14.5
800.......	4.2	4.8	5.3	6.1	....	6.4	7.0	8.5	10.8
900.......	2.0	2.9	3.8	5.0	....	2.9	3.4	4.7	7.8

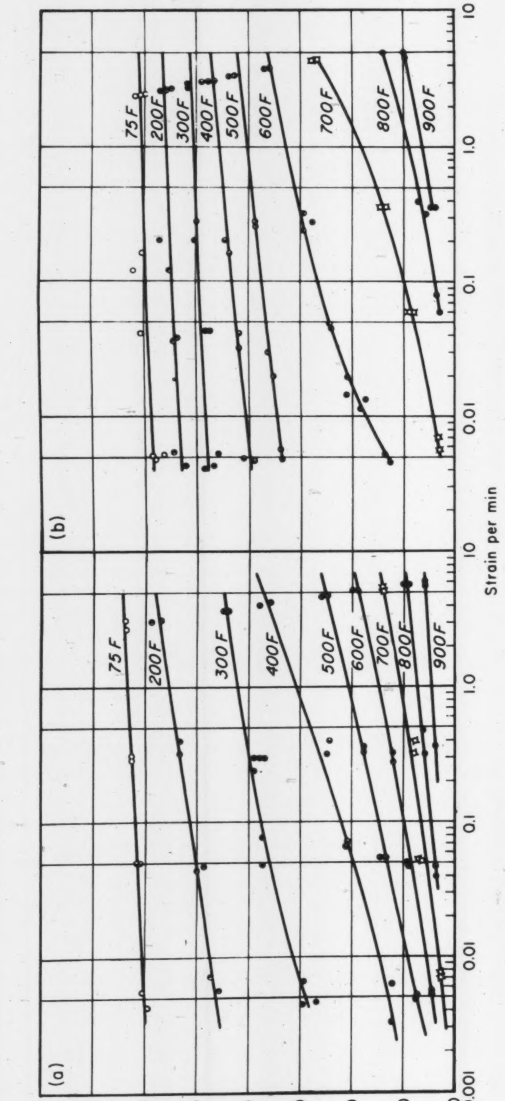
TABLE IV.—*Concluded.*

Test Temperature, deg Fahr	Tensile Yield Strength, 1000 psi				Tensile Strength, 1000 psi				
	Strain per min				Strain per min				
	0.005	0.050	0.50	5.0	0.005	0.050	0.10	0.50	5.0
HZ32A-T5 Alloy									
75.......	14.5	14.5	14.5	14.5	....	32.0	32.0	32.0	32.0
200.......	12.5	13.0	13.4	13.8	....	24.3	24.7	25.6	27.0
300.......	10.5	11.3	12.0	12.7	....	19.7	20.2	21.3	23.0
400.......	9.0	9.9	10.9	11.9	....	16.0	16.5	17.7	19.5
500.......	7.3	8.2	9.1	10.3	....	12.5	13.0	14.0	15.5
600.......	6.6	6.8	7.3	8.9	....	10.8	11.0	11.5	12.1
700.......	6.0	6.1	6.5	7.8	....	10.2	10.4	10.6	11.0
800.......	4.8	5.1	5.5	6.0	....	7.1	7.5	8.3	9.4
900.......	2.5	3.2	3.9	4.5	....	3.9	4.1	5.0	6.8
ZH62A-T5 Alloy									
75.......	27.5	27.5	27.5	27.5	....	41.5	41.5	41.5	41.5
200.......	23.2	24.3	25.4	26.5	....	33.4	33.9	35.1	36.9
300.......	19.5	21.2	22.9	24.5	....	25.7	26.7	28.7	31.8
400.......	14.5	17.0	19.4	21.8	....	18.4	19.2	21.5	26.0
500.......	9.7	11.7	14.0	17.5	....	12.8	13.5	15.6	20.5
600.......	7.5	8.6	10.2	13.4	....	9.5	10.1	11.7	15.6
700.......	6.0	7.0	8.4	11.0	....	7.5	8.0	9.4	12.0
800.......	3.9	4.8	6.1	8.4	....	5.0	5.5	6.7	9.0
900.......	1.5	2.0	3.0	4.9	....	2.5	2.6	3.2	5.0

extensometer (model T2-M, ½ in. gage length) was used in the test program. The stress-strain recorder was used in the tests at strain rates of 0.005, 0.05, and 0.5 per min. For the fastest tests at a strain rate of approximately 5.0 per min (obtained with a crosshead speed of 20 in. per min) an oscillograph, equipped with a Polaroid Land camera, was used to record the data. A stress signal received from a 10,000-lb capacity SR-4 load cell was preamplified with an Ellis Associates d-c amplifier (model 1010) and applied to the *y*-axis amplifier of the oscillograph. The stress signal was obtained by adjusting the excitation voltage to the load cell with a variable resistor to correspond to the cross-sectional area of the specimen. A strain signal from the Microformer extensometer, excited by an audio oscillator, was directly applied to the *x*-axis amplifier of the oscillograph.

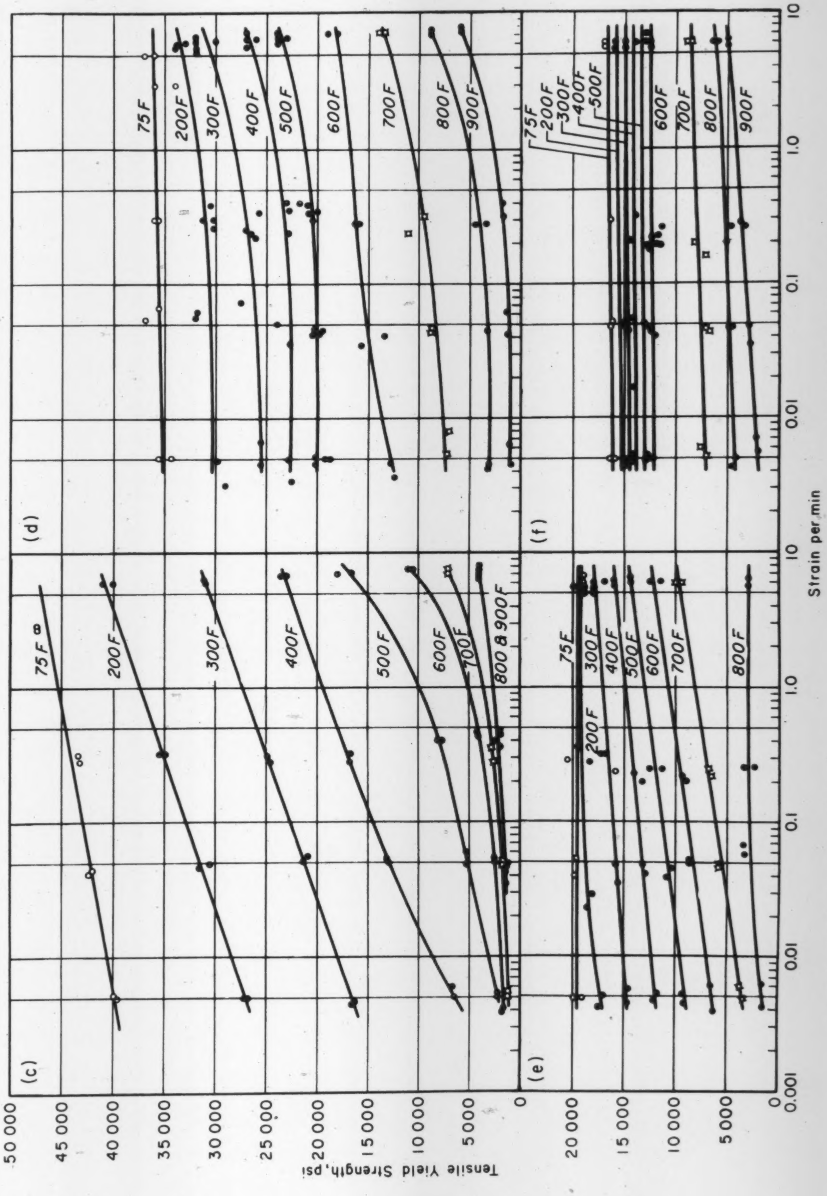
Difficulty was experienced in running some of the tests at strain rates of 0.5 per min. In these tests, the limiting load-response speed of the testing machine was momentarily exceeded, giving inaccurate yield strength values. Questionable values were rechecked using the oscillograph to record the data, thereby eliminating the response speed of the machine weighing system as a source of error.

The strain rates for the three slower test speeds were calculated from time-interval marks produced on load-strain curves by a pen attached to a periodically excited electromagnet. Strain rates for the fastest tests were calculated from



(a) AZ31B-H24 sheet alloy. (b) HK31A-H24 sheet alloy.

FIG. 1.—Effect of Strain Rate on the Tensile Yield Strength of Various Magnesium Alloys.



(c) ZK60A-T5 extrusion alloy.
(e) AZ91C-T6 sand-casting alloy.

(d) HM31XA-F extrusion alloy.
(f) HK31A-T6 sand-casting alloy.

FIG. 1.—(*Continued.*)

stress-time curves. Stress-time curves were obtained by applying the stress signal to the y-axis of the oscillograph while the electron beam was sweeping in the x-direction at a known speed. Knowing the stresses corresponding to given strain increments from stress-strain curves and the elapsed times to reach these stresses from stress-time curves, strain rates were calculated.

Heating to test temperature was accomplished by passing electric current directly through the test specimens. Current was supplied by a 440 v to 0.8–3.0 v step-down transformer and regulated by a Westinghouse Furnatron Control system (FST-2) equipped with a Leeds & Northrup controller (series 60). A 30 gage iron-constantan thermocouple, percussion welded to the center of the specimen with a capacitor-discharge welder, was used to supply a temperature signal to the controller.

The heating time to test temperature was approximately 10 sec and exposure at temperature prior to testing was approximately 5 sec. The controller limited temperature overshoot and fluctuations to ±2 F at the center of the specimen. Measurement of thermal gradients in the specimens showed the temperature at the ends of the gage length to be not more than 10 F below the control temperature.

RESULTS AND DISCUSSION

Tensile yield strength and tensile strength data for each alloy tested were plotted as a function of strain rate and test temperature on semi-logarithmic graphs. Faired curves were drawn through the data points to observe the relationships between variables. To conserve space, strength values obtained from the curves at strain rates of 0.005, 0.050, 0.50, and 5.0 per min are presented in Tables II, III, and IV for the sheet, extrusion, and sand casting alloys, respectively, instead of reproducing all the original curves. Tensile strengths are also given at a strain rate of 0.1 per min because this speed is widely used in industry. Values were obtained from the curves instead of tabulating the actual test values because the strain rates obtained experimentally varied randomly from those desired. At a constant crosshead speed for the testing machine, the strain rate over the gage length of the specimen increases with increasing strain, because of localized plastic flow in that region. This is especially evident between the proportional limit and the yield strength. Therefore, to minimize deviation from the desired strain rates, strain pacing was used for the two lower testing speeds rather than maintaining constant crosshead speed. Despite such precautions, however, there remained some variation in strain rate throughout the test. To minimize its effect on results, yield strength data were plotted on the graphs at the average strain rate between the proportional limit and the yield strength, and tensile strength data were plotted at the average strain rate between the yield strength and the tensile strength.

The yield strengths and tensile strengths of many magnesium alloys increase with increase in strain rate. These alloys are considered to be strain-rate sensitive. Some of the alloys, on the other hand are almost completely insensitive to strain rate at certain temperatures.

For purposes of illustration, three lower-temperature alloys, AZ31B-H24, ZK60A-T5, and AZ91C-T6, and three elevated-temperature alloys, HK31A-H24, HM31XA-F, and HK31A-T6, were selected to demonstrate the general effects of strain rate on the yield strength of magnesium alloys. These effects for the sheet alloys (AZ31B-H24 and HK31A-H24), extrusion alloys (ZK60A-T5 and HM31XA-F), and sand-casting alloys (AZ91C-T6 and HK31A-T6) are shown in Fig. 1. The yield strengths of two lower-temperature alloys, AZ31B-H24 and ZK60A-T5, shown in Figs. 1 (*a*) and (*c*), have gradually increasing sensitivity to strain rate with increasing temperature up to 400 F, where maximum sensitivity is observed. HK31A-H24 and HM31XA-F, the elevated-temperature alloys shown in Figs. 1 (*b*) and (*d*), exhibit the same type of behavior but have maximum sensitivity at 700 F. The lower-temperature casting alloy AZ91C-T6 (Fig. 1 (*e*)) shows a gradual increase in sensitivity to strain rate, up to 700 F. However HK31A-T6 (Fig. 1 (*f*)) shows practically no strain-rate sensitivity at temperatures up to 600 F.

The other alloys tested in the program did not all respond to strain rate according to any of the patterns shown by the examples. In general, however, properties of cast alloys are less sensitive to changes in strain rate than are wrought alloys in the same alloy system. The reader is directed to the tables for strength data on other alloys of interest.

It is important to realize that large variations in strength may be obtained if strain rate is not adequately controlled. For example, referring to Fig. 1 (*b*), when HK31A-H24 sheet is tested at 600 F at strain rates of 0.003 and 0.007 per min, the yield strength varies approximately 2000 psi as a result of the difference in strain rate alone. However, this range of variation in strain rate is within the range of scatter expected if a strain rate of 0.005 per min is desired but the tests are not strain paced. Therefore some dependable means for accurate control of strain rates is necessary to obtain consistent results, especially when testing at temperatures where a high strain-rate sensitivity occurs in the material. It would also be highly desirable to have standardized strain rates included in both testing and material specifications in order to (*a*) ensure uniformity of test methods used in material certification or capability tests, (*b*) allow direct comparison of data determined at different laboratories on the same alloys, and (*c*) allow structural designers to compare competitive materials under identical conditions. The selection of a standardized strain rate should take into account regions of high and low strain-rate sensitivity. Thus, although the material may be used under conditions where high-strain-rate sensitivity is encountered, it would be better to specify elevated-temperature properties in procurement specifications at temperatures which do not produce high strain-rate sensitivity. At these temperatures testing problems due to strain-rate control are minimized and material quality can be more accurately evaluated.

Dotson, Kattus, and Morrison[3, 4] have previously investigated the effect of strain rate and temperature on the strength of some materials, including the magnesium alloys AZ31A-H24 and ZH62A-T5. The trends shown in their work for these alloys are generally the same as the trends observed in this investigation.

CONCLUSIONS

Significant differences in the indicated strength of magnesium alloys with variations in testing speed are demon-

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[3] C. L. Dotson and J. R. Kattus, "Tensile Properties of Aircraft-Structural Metals at Various Rates of Loading After Rapid Heating," Part 1, *Technical Report 55-199*, Wright Air Development Center, Dayton, Ohio.

[4] J. D. Morrison and J. R. Kattus, "Tensile Properties of Aircraft-Structural Metals at Various Rates of Loading After Rapid Heating," Part 2, *Technical Report 55-199*, Wright Air Development Center, Dayton, Ohio.

strated in this study. In view of the magnitude of these differences, the data presented should be useful to structural designers. The data also demonstrate the need for adequate control of strain rate to obtain consistent test results. Strain rate requirements in test procedures and material specifications should be standardized so that uniform test methods will be employed. To minimize testing problems associated with elevated-temperature certification or capability testing, specification temperatures corresponding to low strain-rate sensitivity in the material should be selected.

AGING CHARACTERISTICS OF NICKEL-CHROMIUM ALLOYS CONTAINING APPRECIABLE AMOUNTS OF TITANIUM AND ALUMINUM *

By Neil E. Rogen[1] and Nicholas J. Grant[1]

Synopsis

This investigation was concerned with the aging characteristics and high-temperature strength properties of vacuum melted, wrought nickel-chromium-titanium-aluminum alloys. The aluminum plus titanium content of these simple alloys varied from four to six weight per cent (6.4 to 8.8 atomic per cent) and the titanium to aluminum ratio (atomic) varied from 1.0 to 1.6. Phase identification, particle size and lattice parameter measurements were made on electrolytically extracted precipitates. The variation of hardness with aging temperature and time was studied and correlated with the particle size data. Finally, the stress rupture properties were measured, and an attempt was made to relate the high-temperature strength properties to the aging characteristics exhibited by these alloys.

One important group of alloys that has been developed to meet the requirements of elevated temperature service is that based on nickel-chromium and age hardened with titanium plus aluminum. Originally based on the simple system of four elements, much more complex alloys have been developed which contain up to 30 per cent cobalt, up to 10 per cent molybdenum, up to 0.15 per cent carbon, and some columbium. These additions, along with the use of vacuum melting and casting, which in turn permitted higher titanium plus aluminum additions, have resulted in an increase in service temperature of about 100 to about 1600 F.

It has been determined **(1–4)**[2] that the properties of the simple nickel-chromium-titanium-aluminum alloys are dependent on the precipitation and distribution of intermetallic phases and carbides and nitrides. Nordheim and Grant **(1)** have shown that the titanium plus aluminum content and the titanium-to-aluminum ratio has an important effect on the mechanical properties. They concluded that stress rupture strength at 1500 F was improved by increasing the titanium plus aluminum content to 4 weight per cent and by increasing the titanium to aluminum ratio.

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* Presented at the Sixty-first Annual Meeting of the Society, June 22–27, 1958.

[1] Research Assistant and Professor, respectively, Department of Metallurgy, Massachusetts Institute of Technology, Cambridge, Mass.

[2] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 708.

Taylor and Floyd (5) have determined the nickel-rich end of the nickel-chromium-titanium-aluminum constitution diagram. This work has led to an understanding of the phase relationships present in the Nimonic type alloys. The only two precipitating phases present are the ordered face-centered-cubic Ni_3Al (γ') and the hexagonal Ni_3Ti (η). They can both exist in equilibrium with the nickel-rich solid solution. Unlike Ni_3Ti, the Ni_3Al has a considerable range of solid solubility. In Ni_3Al a maximum of three out of every five aluminum atoms can be replaced by titanium, which results in a lattice parameter increase of approximately 0.5 per cent.

TABLE I.—CHEMICAL ANALYSES OF THE EXPERIMENTAL ALLOYS.

COMPOSITION, WEIGHT PER CENT

Alloy	Chromium	Titanium	Aluminum	Carbon	Manganese	Silicon	Nickel[a]
8v	20.71	2.98	1.43	0.04	...	...	74.79
10v	19.30	3.07	2.09	0.03	0.05	0.004	75.46
11v	19.78	3.85	1.38	0.03	...	...	74.91
12v	18.89	2.89	3.12	0.04	...	...	75.01
13v	18.90	3.97	2.05	0.03	...	...	75.00

COMPOSITION, ATOMIC PER CENT

Alloy	Chromium	Titanium	Aluminum	Carbon	Nickel	Titanium + Aluminum	Titanium ÷ Aluminum
8v	22.06	3.47	2.95	0.18	71.06	6.42	1.18
11v	21.11	4.46	2.84	0.14	70.86	7.30	1.57
13v	20.15	4.59	4.21	0.14	70.85	8.80	1.09

[a] Nickel content by difference.

Nordheim and Grant (1), Brockway and Bigelow (6), and Wilde and Grant (2) have all determined that the precipitation of $Ni_3(Al, Ti)$ (γ') phase was responsible for the hardening both in the more simple Nimonic alloy and in the more complex alloys, except possibly M-252, which contains about 10 per cent molybdenum (2).

Ni_3Al is a face-centered-cubic ordered structure with a lattice parameter of 3.560 Å. This phase forms at 1395 C and is the product of a peritectic reaction between the melt and NiAl (5). Aluminum atoms occupy the cube corners and nickel occupies the cube faces of the unit cell. When titanium substitutes for aluminum it does so preferentially. Ni_3Ti is an ordered, close-packed hexagonal intermetallic phase which exists at the near stoichiometric ratio. The lattice parameters are a = 2.5454 kx and c = 8.2900 kx; the melting point is 1370 C. At low aging temperatures, this phase forms a pearlitic structure while at high temperatures it forms a Widmanstätten precipitate with the basal (00.0) planes in congruence with the (100) planes of the matrix.

The aim of the investigation was to extend the study of the aging characteristics and mechanical properties of simple nickel-chromium-titanium-aluminum alloys containing titanium plus aluminum in excess of 4 weight per cent (1). Specifically, variations in hardness, particle size, and rupture properties with variations in titanium and aluminum content, temperature, and stress were studied. It was hoped that relationships could be found between these many variables, since an understanding of these relationships would aid in the future design of improved high-temperature materials.

Materials and Experimental Procedures

All the alloys investigated were received as vacuum melted and cast and forged bar stock of $2\frac{1}{4}$-in. diameter. They were then reforged to $\frac{3}{8}$-in. diameter. Chemical analyses of the bars are given in Table I.

Alloys 8v, 10v, and 11v were easily forged to $\frac{3}{8}$ in. in diameter. Alloy 13v was forged with considerable difficulty to $\frac{1}{2}$ in. in diameter, and 12v cracked during forging. The forging temperature was 2200 F and was not allowed to drop below 1900 F.

All alloys subjected to stress-rupture testing were given identical heat treatments. While six different heat treatments were used for each alloy, those treated at 2250 F and water quenched were too brittle to test. The heat treatments were as follows:

1. Solution at 1900 F, 5 hr; oil quenched; aged at 1400 F, 24 hr.
2. Solution at 1900 F, 5 hr; oil quenched; aged at 1600 F, 2 hr.
3. Solution at 2000 F, 4 hr; water quenched; aged at 1400 F, 24 hr.
4. Solution at 2000 F, 4 hr; water quenched; aged at 1600 F, 2 hr.
5. Solution at 2250 F, 2 hr; water quenched; aged at 1400 F, 24 hr.
6. Solution at 2250 F, 2 hr; water quenched; aged at 1600 F, 2 hr.

The solutioning and aging temperatures that were selected produced significant variations in alloy structure. These variations enabled some correlation to be made between structure and strength properties. Aging times were used which caused the alloys to be aged to approximately peak hardness. The solution treatment at 2250 F produced severe grain growth in all alloys; on the other hand, at 1900 F the precipitate in the higher aluminum plus titanium content alloys did not completely dissolve. At 1900 and 2000 F the solution treatment produced the same grain size in all alloys (ASTM Nos. 4 and 5).[3]

X-ray and Rockwell hardness measurements were used to follow the aging process. Phase identification, parameter measurements, and particle sizes were determined by X-ray techniques.

All hardness measurements were made on $\frac{1}{4}$-in. slices from the reforged bar stock, which had been water-quenched from the aging temperature. One of the flat sides was prepared for metallographic examination prior to taking Rockwell measurements. Hardness values are an average of at least six measurements, each measurement being within ±2 units of the average value.

An electrolytic polishing and etching technique was used for metallographic examination of these alloys, the reagent being a solution of H_3PO_4 saturated with chromium trioxide. A potential of 30 v was used for polishing and 15 for etching.

It was reasonably certain that the only precipitating phase present in these vacuum melted nickel-chromium-aluminum-titanium alloys was the ordered $Ni_3(Al, Ti)$ structure. The observed values of the matrix and the precipitate parameters were usually within 0.5 per cent of one another. Therefore, identification of this phase would depend on resolving the simple cubic reflections (100), (110), (210), etc. Since accurate parameter measurements would require clear differentiation between matrix and precipitate lines, it was necessary to concentrate the precipitate by extraction techniques in order to intensify the precipitate diffraction lines.

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[3] Classification of Austenite Grain Size in Steel (E 19 – 46), 1955 Book of ASTM Standards, Part 1, p. 1602.

TABLE II.—PHASE IDENTIFICATION—CuK α-RADIATION.

hkl	Ni_3Al	Ni_3Al[a] Standard[b]		$Ni_3(Al_{0.5}, Ti_{0.5})$[a] Standard	
	d calculated, Å	*d* observed, Å	*I*	*d* observed, Å	*I*
100.....	3.56	3·60	w	3.60	w
110.....	2.52	2.55	w	2.54	w
111.....	2.06	2.07	s	2.07	s
200.....	1.78	1.80	ms	1.80	s
220.....	1.26	1.27	m	1.26	m
331.....	1.07	1.08	m	1.08	s
222.....	1.03	1.03	w	1.04	mw
400.....	0.87	0.87	vw	0.90	mw
331.....	0.82	0.82	mw	0.82	m
420.....	0.80	0.80	ms	0.80	m

hkl	Alloy 8v[c]		Alloy 11v[c]		Alloy 13v[c]	
	d observed	*I*	*d* observed	*I*	*d* observed	*I*
100.....	3.60	vw	3.63	vw	3.58	w
110.....	2.54	vw	2.54	vw	2.53	w
111.....	2.07	vs	2.08	vs	2.07	vs
200.....	1.80	s	1.80	s	1.80	s
220.....	1.27	m	1.27	m	1.27	m
311.....	1.08	m	1.08	m	1.08	m
222.....	1.03	w	1.04	w	1.03	w
400.....	0.90	w	0.90	w	0.90	w
331.....	0.82	m	0.82	m	0.82	m
420.....	0.80	m	0.80	m	0.80	m

[a] Same standards—Wilde and Grant (**2**).

[b] Simple cubic lines (210) and (211) were resolved at 1.59 and 1.45, respectively. Intensities were w and vw.

[c] Alloys 8v, 11v, 13v—solutioned 1900 F 5 hr, oil-quenched, aged 24 hr 1400 F.

The technique used was basically the same as that described by Wilde and Grant (2). The electrolytic cell consisted of a bar specimen of the alloy as the anode, and a 3 sq in. tantalum sheet encased in cellophane dialysis tubing as the cathode. A 400-ml beaker containing 350 ml of a 15 per cent H_3PO_4 solution completed the cell. The current was 0.5 amp per sq in. and was supplied by a storage battery. The extracted precipitate particles were centrifuged, decanted, washed and dried, carefully repowdered, and then prepared for X-ray analysis.

Lattice parameter measurements were made using a General Electric RDX Spectrogoniometer and filtered chromium radiation. The (220) line was measured and corrected with a silicon standard. All values are an average of at least four measurements, and all measurements were within ±0.001 Å.

Specimens selected for particle size determination and lattice parameter measurements received the same heat treatment as the stress rupture specimens. This allowed the precipitate particle size of stress rupture specimens to be established before testing.

Particle sizes were determined from the electrolytically extracted residues. However, since any dissolution of the intermetallic compound during the extraction would result in an inaccurate size determination, it was necessary to examine this technique thoroughly.

The susceptibility of the extracted residue to electrolyte attack was determined by exposing the residue to a 15 per cent H_3PO_4 solution for two weeks, measurements showed that no attack had taken place. If it is assumed that all of the residue is the $Ni_3(Al, Ti)$ intermetallic compound and that all of the aluminum and titanium is precipitated, then a comparison of the weight of the residue to the weight of the alloy dissolved would allow an evaluation of the extraction technique. In three separate tests the weight of residue came within 2 per cent of the computed value. Furthermore, chemical analysis of the used electrolyte showed that only negligible amounts of aluminum and titanium were in solution. On the basis of these results, it was felt that the extrac-

TABLE III.—LATTICE PARAMETERS OF PHASES PRESENT IN ALLOYS 8v, 11v, 13v, SOLUTIONED 1900 F, 5 HR, OIL-QUENCHED, AGED 24 HR AT 1400 F.

CrK α (220) Line Corrected with Silicon Standard

Alloy[a]	Matrix, Å	Precipitate, Å	Misfit, per cent	Rockwell Hardness, C Scale
8v..........	3.569	3.592	0.65	40
11v.........	3.572	3.590	0.50	43
13v.........	3.572	3.588	0.45	43

[a] The metallic radii of titanium, aluminum, chromium, and nickel are given by Pauling as 1.467, 1.429, 1.267, and 1.244 Å, respectively.

TABLE IV.—PARTICLE SIZE AS MEASURED BY X-RAY LINE BROADENING FOR ALLOYS AGED TO APPROXIMATELY PEAK HARDNESS.

Alloy	Solutioning Treatment	Aging Treatment	Average Particle Size, Å	Average Particle Spacing, Å
8v.............	1900 F, 5 hr, OQ[b]	1400 F, 24 hr	205	85[a]
	1900 F, 5 hr, OQ	1600 F, 2 hr	390	165
	2000 F, 4 hr, WQ[c]	1400 F, 24 hr	265	115
	2000 F, 4 hr, WQ	1600 F, 2 hr	350	150
11v.............	2000 F, 4 hr, WQ	1400 F, 1 hr	90	...
	1900 F, 5 hr, OQ	1400 F, 24 hr	595	200
	1900 F, 5 hr, OQ	1600 F, 2 hr	870	290
	2000 F, 4 hr, WQ	1400 F, 24 hr	290	95
	2000 F, 4 hr, WQ	1600 F, 2 hr	390	130
13v.............	1900 F, 5 hr, OQ	1400 F, 24 hr	870	210
	1900 F, 5 hr, OQ	1600 F, 2 hr	940	220
	2000 F, 4 hr, WQ	1400 F, 24 hr	460	110
	2000 F, 4 hr, WQ	1600 F, 2 hr	490	115

[a] This is the calculated interparticle spacing. The shortest distance between particles was calculated assuming perfect particle distribution and complete precipitation of all solute atoms, and a cubic particle shape.

[b] OQ = oil-quenched.

[c] WQ = water-quenched.

tion technique was adequately quantitative.

Particle size was determined by X-ray line broadening. Filtered CrK α-radiation was used and the half breadth of the (111) line was measured for line broadening. The silicon (331) line was used as a standard.

Specimens having 0.250-in. diameter and 1-in. gage length were used for all creep-rupture tests. All tests were carried out at 1500 F. The maximum time required for bringing the specimen up to temperature was ¾ hr. Temperature control was within 2 F of the indicated test temperature.

RESULTS AND DISCUSSION

X-ray patterns from extracted residues of alloys 8v, 11v, and 13v were nearly identical to those made from a Ni_3(Al, Ti) standard. The similarity among observed reflections, *d* spacings, and intensities confirmed that the only precipitating phase present in these alloys is the ordered intermetallic, face-centered-cubic (Ni_3Al, Ti).

Table II shows the various X-ray patterns and their corresponding intensities. Only the first two simple cubic lines (100) and (110) were resolved for all the Ni_3(Al, Ti) patterns. This is due to the relative scattering factors of titanium and aluminum and to the fact that titanium and aluminum occupy the corners of the face-centered-cubic lattice. Because the scattering factor is higher at all angles for titanium, the simple cubic lines are weaker when the titanium to aluminum ratio is high. A good example of the intensity variation with the titanium to aluminum ratio can be seen by comparing either the (100) or (110) reflections of alloys 8v and 11v (vw) to alloy 13v (w). Alloy 13v has a lower titanium to aluminum ratio than either alloy 8v or 11v.

Matrix parameter and extracted residue precipitate studies were made using the same specimens. Matrix measurements were taken on the electrolytically polished surface of the solid specimens. Chromium radiation was used instead of copper radiation because it produced a lower fluorescent background.

Table III shows that alloy 8v has a smaller matrix lattice parameter than alloy 11v or 13v and that the lattice parameter of the $Ni_3(Al, Ti)$ decreases from 8v to 11v to 13v. Under equilibrium conditions, considering metallic radii (7) the lattice parameter of the $Ni_3(Al, Ti)$ precipitates should increase with increasing titanium to aluminum ratio, and the matrix lattice parameters should decrease with increasing nickel content. At equilibrium, therefore, precipitates in alloy 11v should have the largest lattice parameter, and the matrix lattice parameter should decrease from 8v to 11v to 13v. Since this is not the case, it can only be concluded that equilibrium conditions are not met by aging for 24 hr at 1400 F.

The lattice parameter of the ordered face-centered-cubic $Ni_3(Ti, Al)$ is affected by many factors, among which are the aging temperature and time, chemical composition, metallic radii, and lattice positions, and the diffusion rates of titanium and aluminum. (Titanium diffuses at a slower rate than aluminum.) Because of these many contributing factors, it is most difficult to explain the parameter value resulting from the nonequilibrium conditions of heat treatment.

The degree of misfit between matrix and precipitate, and the hardness are shown in Table III. They are similar in magnitude to measurements reported for Inconel 550 (2) and for the simple nickel-chromium-titanium-aluminum alloys (1).

TABLE V.—HARDNESS AND RELATED DATA FOR EXPERIMENTAL ALLOYS.

Alloy Solution Temperature....	Solution Rockwell Hardness, C Scale		
	1900 F	2000 F	2250 F
8v	21	11	47 R_a
10v	25	...	16
11v	33	22	21
13v	36	32.5	31

ROCKWELL HARDNESS, C SCALE, OF OVERAGED ALLOYS AGED AT 1600 F FOR 336 HR

Alloy Solution Temperature....	1900 F	2250 F
8v	18	16
11v	25	23
13v	25	24

ROCKWELL HARDNESS, C SCALE, OF BULK STANDARD SAMPLES

Ni_3Al	12
$Ni_3(Al_{0.5}Ti_{0.5})$	31

VOLUME OF PRECIPITATE[a]

8v	25.7
10v	30.7
11v	29.2
13v	35.2

[a] As calculated assuming complete precipitation and little solid solubility.

In the nickel-chromium-titanium-aluminum system, the following variables importantly affect the particle size of the $Ni_3(Al, Ti)$:

1. Solution treatment;
2. Aging time and temperature;
3. Aluminum plus titanium content, and aluminum to titanium ratio; and
4. Composition of the matrix.

Solution treatment has a profound effect on the particle size on aging. In a partially solutioned material, the average particle size will be larger than that of a fully solutioned specimen of the same material after the same aging treatment. This is due to the existence of many small particles in the partially solutioned material that act as nuclei or growth points. The precipitating phase will form on these particles and in this manner raise the average particle size above that of the fully solutioned and aged specimens. This is demonstrated in Table IV. Alloys 11v and 13v are fully solutioned at 2000 F and only partially at 1900 F; electrolytic digestion of these alloys after the 2000 F solution treatment showed essentially no residue. This variation in solution treatment results in an average particle size difference of approximately 400 Å.

Alloy 8v is an example of a material which is slightly in the two phase field at 1900 F and wholly in the single phase field at 2000 F. This is in contrast to alloys 11v and 13v which are deep in the two phase field at 1900 F. It is likely that the structure of alloys solution treated at 1900 F differs in the coarseness of the undissolved precipitate particles. The undissolved particles in alloy 8v should be the finer particles. Hardness values of 21 Rockwell C and 11 Rockwell C were measured for 8v solution treated at 1900 F and 2000 F, respectively. (See Table V.) This difference in hardness is due to the presence of undissolved precipitate particles in the 1900 F solutioned specimen. The change in grain size was small between the 1900 and 2000 F treatments. During 1400 F short-time aging the structure that contained fine particles would precipitate additional fine particles due to the relatively depleted matrix of the incompletely solutioned structure. A comparison of the average particle size of the differently solutioned but identically aged structure should show a larger average particle size for the completely solutioned structure. The particle size variation of alloy 8v aged at 1400 F illustrates this point. By aging 8v at a higher temperature, this particle size difference is eliminated.

Following a given solution treatment, aging at 1600 F for 2 hr resulted in a larger particle size than aging at 1400 F for 24 hr, and both aging treatments gave coarser particles than aging for 1 hr at 1400 F. As expected, average particle size increased with increasing temperature and time.

Particle size increased with increasing titanium plus aluminum content and decreased with increasing titanium-aluminum ratio. Nordheim and Grant (1) and Wilde and Grant (2) observed the same relationship of particle size to titanium-aluminum ratio. Total titanium plus aluminum content in these alloys increased from 8v to 11v to 13v and the titanium-aluminum ratios were 1.18:1, 1.57:1 and 1.09:1, respectively. Alloys 8v and 13v have approximately the same titanium-aluminum ratio but differ considerably in titanium plus aluminum content. This should and does result in a sizeable difference in particle size after the identical heat treatments. Alloy 11v has a much larger titanium-aluminum ratio than either 8v or 13v and has a titanium plus aluminum content that falls between those of 8v and 13v. The slower diffusion rate of titanium results in a smaller particle size than would be expected if only the titanium plus aluminum content were considered.

The particle shape of $Ni_3(Al, Ti)$ in alloys of chemical compositions similar to 8v, 11v, and 13v were found to be cubic by Corey, Rowe, and Freeman (8). They also found that the sites of precipitation were controlled by the quenching rate. Particle distribution was generally uniform with some concentration at grain boundaries. During this investigation the quenching procedure was kept constant for each solutioning temperature to eliminate any variation in the mode of precipitation.

Coherency strains tend to increase the diffusion rate around the coherent particle. An increase in misfit results in a local increase in diffusion rate. In these alloys it is evident that coherency strains have little if any influence on particle size. Alloy 8v has the greatest misfit and the smallest particle size for identical heat treatments. This is contrary to what would be expected if coherency strains had a primary effect on particle size.

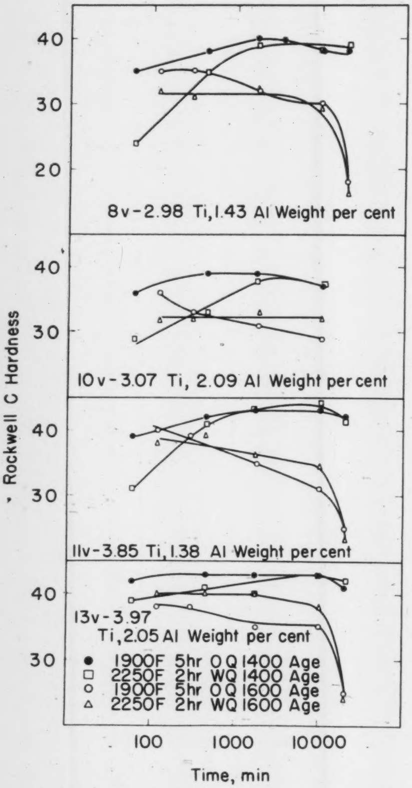

FIG. 1.—Change in Hardness of Alloys 8v 10v, 11v and 13v During Aging at 1400 F and 1600 F.

In general, there are three variables that contribute to the hardness of the age hardening nickel-chromium-titanium-aluminum alloys—the relative quantities of the matrix and the precipitate, the hardnesses of the two phases, and the particle size, distribution, and spacing.

The hardness of the precipitate phase is considerably greater than that of the matrix. This is indicated by the hardness of Ni_3Al and $Ni_3(Al_{0.5}Ti_{0.5})$ standard as shown in Table V. The hardness increased from 11 Rockwell C to 31 Rockwell C as titanium was substituted for aluminum up to a 1:1 ratio. An increase in the titanium to aluminum ratio should result in increased hardness, since there is an increase in lattice strain or a valency effect accompanying the substitution of the larger titanium atom for the somewhat smaller aluminum atom. The lattice parameter of Ni_3Al was found to be 3.560 Å and the parameter of $Ni_3(Al_{0.5}Ti_{0.5})$ was 3.586 Å.

Aging for 336 hr at 1600 F produces an overaged structure in all the alloys (Table V). In the overaged condition, particle spacing contributes little to over-all hardness, and only the relative volumes and hardnesses of the two phases need be considered.

The volume of precipitate phase increases from 8v to 11v to 13v. If volume of the precipitate were the only variable, hardness would be expected to increase progressively from 8v through 13v. However, the titanium-aluminum ratio varies among the alloys. Alloys 8v and 13v have nearly the same titanium-aluminum ratio, while alloy 11v has a considerably higher titanium-aluminum ratio. The higher titanium-aluminum ratio results in a greater hardness. As a result the Rockwell hardness of the overaged 11v and 13v is the same, while 8v is considerably softer.

Figure 1 illustrates the variation of

hardness with time at 1400 and 1600 F. These curves serve as a measure of the variation of particle size and spacing with time and temperature. It appears that stability increases with increasing titanium-aluminum ratio and increasing titanium plus aluminum content. The rate of coalescence is decreased by an increase in titanium-aluminum ratio due to the slower diffusion rate of titanium. An increase in total aluminum plus titanium content causes a larger average particle size and hence a lower surface energy per volume per cent. This lowering of surface energy results in a decreased rate of coalescence.

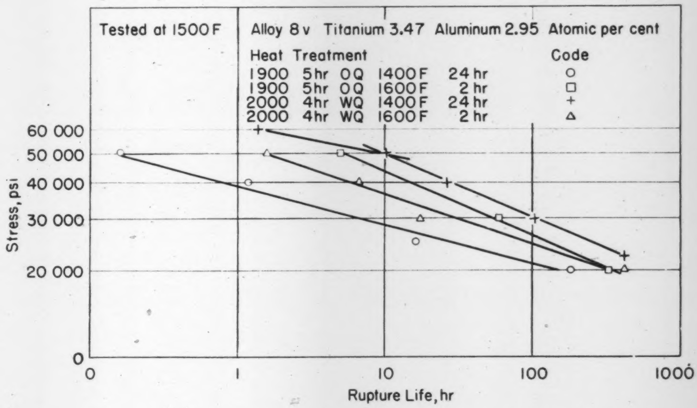

FIG. 2.—Stress *versus* Rupture Life for Alloy 8v, Test Temperature 1500 F.

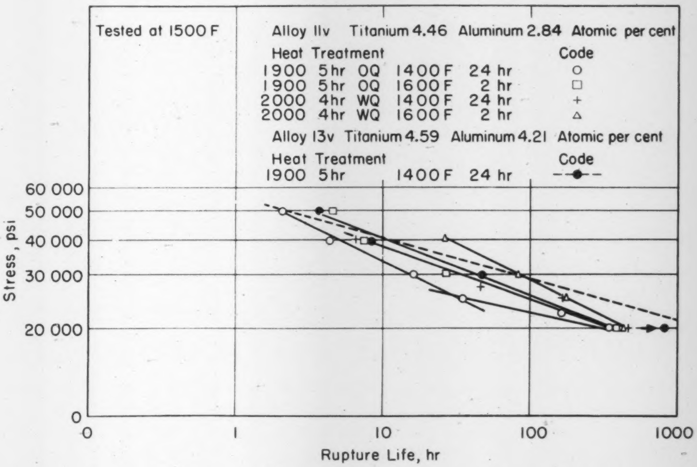

FIG. 3.—Stress *versus* Rupture Life for Alloys 11v and 13v, Test Temperature 1500 F.

Particle sizes and spacings of alloys aged at 1400 F for 24 hr and 1600 F for 2 hr are listed in Table IV. At 1600 F for 2 hr, the alloys are either at peak hardness or somewhat into the overaged region while at 1400 F for 24 hr they are either at or near either side of peak hardness. However, they are all approximately at peak hardness.

TABLE VI.—STRESS RUPTURE STRENGTH AND DUCTILITY VALUES FOR ALLOYS TESTED AT 1500 F.

Alloy	Heat Treated to Approximate Peak Hardness	Stress for Rupture in *n* hours, psi				Approximate Ductility Values at 100 hr, Rupture Life	
		1	10	100	1000	Reduction of Area, per cent	Elongation in 1½ in., per cent
8v	1900 1400	39 000	27 000	20 500	16 000	71	26
	1900 1600	60 000	43 000	27 000	16 500	9	13
	2000 1400	61 000	50 000	30 000	18 500	4	7
	2000 1600	52 000	36 000	24 000	17 000	4	13
11v	1900 1400	56 000	33 000	23 000	19 000	1	8
	1900 1600	65 000	38 000	25 000	18 000	1	15
	2000 1400	52 000	36 000	25 500	17 500	. . .	1
	2000 1600	. . .	50 500	29 000	16 500	. . .	4
13v	1900 1400	55 000	40 000	29 000	22 000	. . .	. . .

At peak or critical hardness, spacings of approximately 200 Å would be expected. Particle spacings listed in Table IV are of this order of magnitude and consistent with the measurements of Brockway and Bigelow (6). These investigators worked with Inconel X and used electron microscopy.

A consistent relationship between particle size and spacing and stress for short-time rupture values could not be found for the nickel-chromium-titanium-aluminum alloys studied in this work. This suggests that particle spacing is not the only, or even the primary, variable that determines the strength properties at elevated temperatures. At 1500 F, deformation is accommodated by grain distortion plus grain boundary sliding. The occurrence of grain boundary movement accounts for some important portion of the total deformation. Therefore, it is unlikely that the strengthening mechanisms as determined by particle spacing alone could explain the strength properties.

Figures 2 and 3 show the stress *versus* rupture time plots for alloys 8v, 11v, and 13v at 1500 F. The curves are normal in all respects except for the one for alloy 11v which was solutioned at 1900 F and aged at 1400 F. This curve shows a change in slope, with an upward break. An upward break would suggest strengthening, or an increase in structure stability. Since metallographic examination showed no evidence of grain coarsening during testing, this is not the cause of the upward break. Instead, it appears that the 1900 F solution treatment followed by 1400 F aging results in a partially aged structure in which further active aging is taking place in the short-time tests. Once the structure is fully aged, the slope achieves a normal level.

At 1500 F, at the stresses investigated, intercrystalline cracking was prevalent in all the alloys. Figures 2 and 3 and Table VI summarize the 1500 F strength values for fixed rupture times; and Table VI lists the approximate ductility values for a 100-hr rupture life. Even though there was insufficient material to test alloy 13v fully, it is clear that it has the highest stress value for 1000 hr life at 1500 F. Large differences in strength and ductility are exhibited by these simple alloys with changes in solution and aging temperatures.

Furthermore, the better treatments for alloy 8v are not the best for alloys 11v or 13v. An increase in total content of titanium plus aluminum is seen to be significant in raising the long-time performance of these alloys (see Table V). Unfortunately, the data on particle size and spacing do not contribute to an understanding of the heat treatments because of the lack of information of the stability of the structures during rupture testing.

It is fairly clear, in comparing these results with data on commercial alloys of the same class containing molybdenum, cobalt, columbium and carbon, that the commercial alloys are more stable and predictable than are these simple nickel-chromium-titanium-aluminum compositions (2).

SUMMARY

In simple nickel-chromium-titanium-aluminum alloys containing from 4 to 6 weight per cent titanium plus aluminum, having titanium to aluminum ratios of less than 1.6, there is only one precipitating phase present. This phase is the intermetallic compound $Ni_3(Al, Ti)$, γ'-phase. The precipitation of the γ'-phase was found to be responsible for the high strengths exhibited by these alloys. These results are in agreement with those of Nordheim and Grant (1).

Electrolytic extraction was found to be an effective technique for the near quantitative separation of the precipitate from the matrix. With this technique the amount of precipitating phase can be determined gravimetrically and particle size may be measured by X-ray methods.

Creep-rupture properties are sensitive to the solution treatment used in that partial solutioning has an important effect on the slope of the resultant log stress *versus* log rupture time plot. Changes in the aging structure during test result in large differences in rupture life.

An attempt was made to correlate particle size, spacing and rupture strength at 1500 F. No consistent relationship was found at any stress level. It would appear that such factors as grain size and stability of the precipitate must be taken into consideration in addition to particle size and spacing before a meaningful parameter can be determined.

Nordheim and Grant (1) reported that at a service temperature of 1500 F it was advantageous to raise the total titanium plus aluminum content to 4.5 per cent (maximum carbon 0.05 per cent). They also suggested selecting a composition corresponding to the titanium-rich end of the homogeneity range for γ'. Raising the titanium to aluminum ratio was found to increase the high-temperature strength and to decrease the coalescence rate of the precipitate.

This investigation dealt with alloys of total aluminum plus titanium contents of from 4 to 6 weight per cent. High-temperature strength was found to increase with increasing solute content, but was affected much less by variations in the titanium-aluminum ratio beyond about 1.1 to 1.

Acknowledgments:

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DISCUSSION

Messrs. Ray W. Guard[1] and Robert E. Keith[1] (*presented in written form*).—This interesting and thought-provoking paper prompts several questions and comments:

1. Taylor's study of the nickel-chromium-aluminum-titanium quaternary system (author's reference (5)) indicated that Ni_3 (Al, Ti) will contain considerable amounts of chromium under equilibrium conditions. Yet the authors were able to account for the observed quantities of residues by assuming complete titanium and aluminum precipitation, and also showed negligible titanium and aluminum remaining in the electrolyte. Were the residues analyzed for chromium content? Such analyses might shed light on the nature of the non-equilibrium conditions which were postulated to explain the lattice parameters. Calculations of particle spacing and volume fraction of precipitate would also be affected somewhat if the precipitate contained chromium.

2. It seems doubtful that coherency strains in general could have much effect on particle growth rates, since the zone of solute depletion around a particle will extend far beyond the range of the coherency strains. This is particularly true in the present case, where the degree of mismatch was so slight (0.5 per cent).

3. Were the Ni_3 (Ti, Al) particles microscopically confirmed to be cubic in shape and randomly dispersed? Westbrook's observations are that they are often spherical or occur in disk-shaped colonies of cubes.

4. What evaluation of the reduction of area and elongation figures (Table VI) would the authors make in view of the extensive intercrystalline cracking?

5. Do the authors have any ideas as to why there is such a large hardness increase on aging in this system, particularly at high solutioning temperatures and lower titanium plus aluminum contents? It is difficult to account for this hardness increase in a system where:

(*a*) Matrix and precipitate are of about the same hardness;

(*b*) Both have the same crystal structure; and

(*c*) The degree of mismatch is very small.

Messrs. N. J. Grant and N. E. Rogen (*authors*).—The work of Wilde and Grant (2) with Inconel X550, an alloy somewhat similar to those investigated during this investigation, showed that residues contain from 2 to 3 per cent chromium. A chromium content of this amount would not change appreciably the magnitude of the calculated particle spacing. It is the relative variations and the general magnitude of the particle spacings that are of significance.

We are inclined to agree that coherency strains do not have much (a primary) effect on particle growth. However, because of the small interparticle spacings, the high secondary

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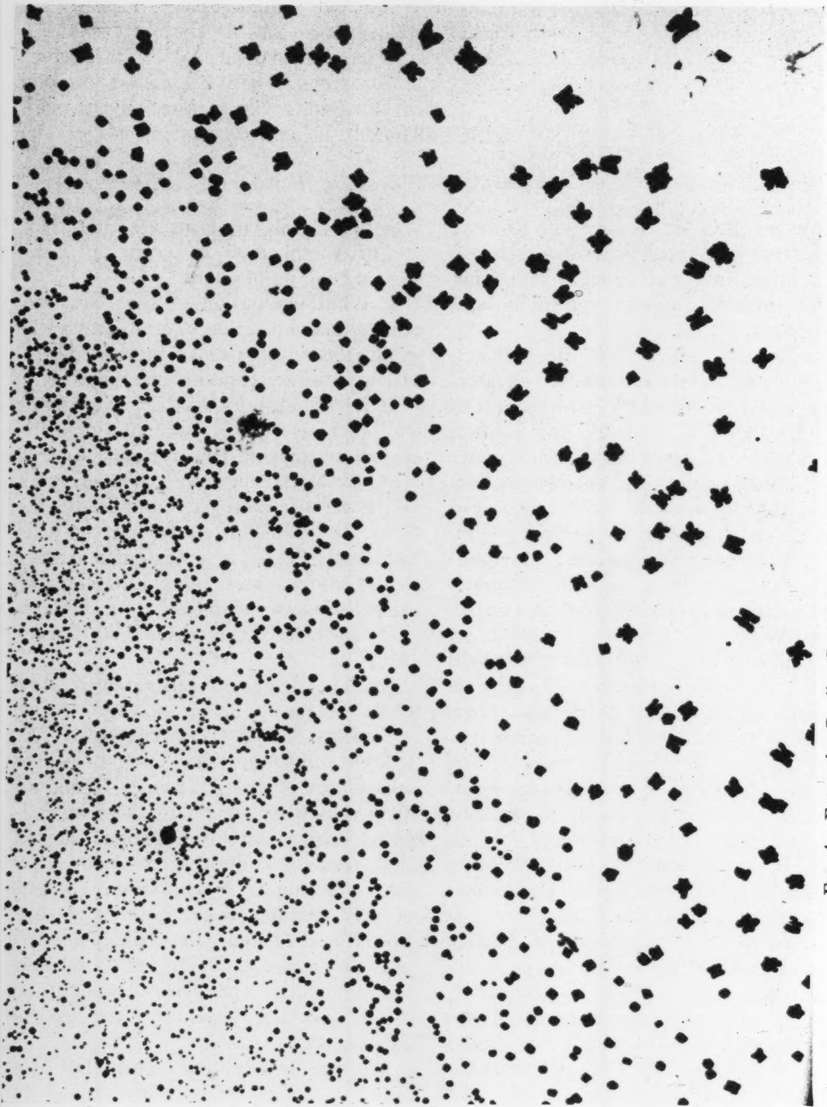

FIG. 4.—Extraction Replica Containing γ' Particles from Nimonic 80A (× 10,000).

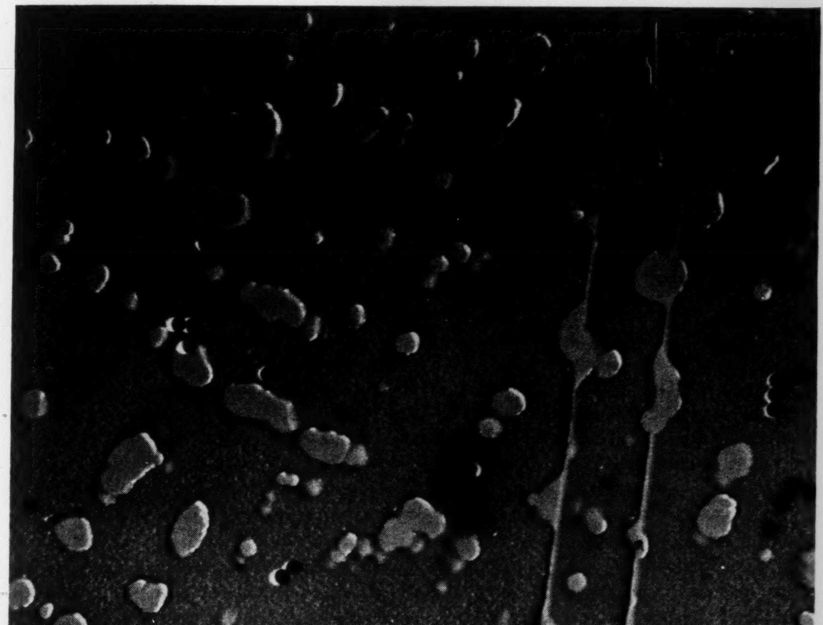

FIG. 5.—Electron Micrograph of Inconel X After 35,000 hr at 1500 F (× 10,000).

phase content, and the lack of knowledge as to the actual extent of coherency strains around coherent precipitate particles, this effect cannot be completely neglected. Work now in progress indicates that subtle variations in the growth rate of precipitate particles may occur during the coherent-incoherent transformation. It is not inconceivable that a variation in the extent of coherency could lead to a variation of the particle growth rate of these alloys.

The work of Corey, Rowe, and Freeman (8) on similar alloys indicated that the precipitate particles are cubic; we assumed this shape as a convenience to calculate interparticle spacing; however, the spacing could be calculated assuming a spherical shape and would not cause a significant change in the spacing relationships.

All the alloys exhibited intercrystalline failure. These alloys exhibit low elongation in tests greater than about 10 hr, have no first-stage creep to speak of, and owe much of the actual recorded elongation values to opening up of intercrystalline cracks during third stage creep.

The variation in hardness during aging can be related to the interparticle spacing. The interparticle spacing in these alloys varies from approximately 100 to 300 Å. The dispersion hardening theories of Orowan, Mott, and Nabarro and Tisher, Hart, and Pry are in general agreement on the degree of dispersion necessary to produce maximum interference with the movement of dislocations.[2] This spacing requirement has been met by the aged alloys of this system.

There is a large difference in hardness between the supersaturated matrix and the precipitate. A bulk sample of Ni_3 (Al, Ti) shows a Rockwell C hardness of 31 while a solution heat treatment of alloy 8v at 2250 F will yield a matrix hardness of Rockwell A 47. Hardening of the alloy beyond Rockwell C 30 must then occur as a result of lattice strains in the system on aging. Furthermore, fine precipitates of Ni_3 (Al, Ti) should be harder than bulk Ni_3 (Al, Ti) because the former should have a lower imperfection density.

MR. KENNETH G. CARROLL.[3]—This paper provides a most interesting approach to the fundamental problem of understanding the behavior of modern superalloys. The authors have opened up new avenues for research by their application of techniques which provide vital information on the structure of precipitation-hardened alloys.

I would like to point out the fact that the alloys studied by the authors contained carbon in concentrations not much different from commercial alloys. Therefore it seems probable that considerable carbide formation accompanies the precipitation of γ' phase. The effects of carbide precipitation in grain boundaries may well be of major importance to the stress-rupture properties observed. In particular, the anamolous curve in the authors' Fig. 3 (alloy 11 v) may be an indication of the formation during the rupture test at 1500 F of one of the two carbides to be expected: $Cr_{23}C_6$ or Cr_7C_3.

X-ray line broadening as a means of measuring particle size suffers from the inherent defect of emphasizing the larger particles in a distribution of sizes. In the event that particles of two distinctly different diameters are present, for example 500 Å and 50 Å, the method used will not reveal the presence of the smaller size. An example of this is shown in the accompanying Fig. 4, taken by J. R. Mihalisin on the electron microscope in our laboratory. This is an extraction replica, at an original magnification of 4000, of the precipitating γ' phase in a commercial nickel-chromium alloy containing aluminum and titanium (Nimonic 80A). This structure resulted from solution treatment for 1 hr at 2200 F, followed by aging for 16 hr at 1600 F. The particles are seen to consist of widely different sizes, and the large ones are evidently the "diced cubes" reported by J. B. Newkirk of the General Electric research group.[4] Development of the large "diced cubes" is ascribed to rapid growth on all eight corners of a preexisting cube, under conditions of drastically altered coherency strains between matrix and precipitate. In a precipitation reaction of this nature, X-ray line broadening measurements could be quite misleading unless accompanied by direct observation in the electron microscope.

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[3] Head, Physics Section, International Nickel Co., Bayonne, N. J.

The authors' statements regarding quantitative extraction of the γ' phase seem to imply that near equilibrium is attained during treatment at 1400 or 1500 F for a day or two. One would expect the kinetics of precipitation to be similar in these alloys and in commercial alloys. An example of the sluggishness of the γ-to-γ' transformation is shown in Fig. 5, which is an electron micrograph of a specimen held at 1500 F for 35,000 hr. Not only is the precipitation reaction still proceeding, but the small dot-like particles of gamma prime are transforming to the needles of η-phase, as established by X-ray diffraction. Although this particular alloy is not identical in composition to those studied by the authors, the precipitation reactions can be expected to be similar, and the approach to equilibrium should not be vastly different.

MESSRS. GRANT AND ROGEN.—Particle size measurement by X-ray line broadening results in an arithmetic average of particle sizes. Remeasurements of particles and particle mixtures measured previously with the electron microscope resulted in an agreement with an error of only 10 per cent. Mixtures of different sized particles gave an arithmetic average of particle size.[5] Our average particle size measurements agreed with those made at the University of Michigan[6] using the electron microscope. They also found the precipitate particles to be relatively uniform in size and distribution. Also, up to 1000 hr after aging, the particles remained spherical. This is in agreement with our recent observations using the electron microscope and extracted precipitates. An average particle size measurement is very valuable for this system because it allows an estimate of the interparticle spacing. This spacing or dispersion is related to the metallurgical properties of these alloys (reference (4) of the paper).

Mr. Carroll's Fig. 5 does not represent a "precipitation reaction still proceeding," but a growth and redistribution of particles after precipitation had been completed long before 35,000 hr. Figure 5 may only be showing formation of γ' on the existing η phase? Further, the solubility of γ' phase for titanium is not exceeded by any of the alloys (reference (5) of the paper) in our group.

If carbide formation is the explanation for the upward break on one stress rupture curve for alloy 11v, there appears to be little reason why this upward break did not occur also in 8v and 13v.

The carbides should form and grow in similar fashion for each alloy because the carbon content is fairly constant. We are inclined to believe that the upward break is instead associated with γ' precipitation during the course of testing.

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[4] J. B. Newkirk, "Mechanism of Precipitation in Copper-2.5 per cent Iron Alloy," *Transactions*, Am. Inst. Mining Engrs., Vol. 209, p. 1214 (1957).

[5] L. S. Birks and H. Freedman, "Particle Size Determination from X-ray Line Broadening," *Journal, Applied Physics*, Vol. 17, pp. 687–692 (1946).

[6] W. C. Bigelow, J. A. Amy, and L. O. Brockway, "The Effect of High Temperature Aging on the Development of Minor Phases in an Age-Hardening Nickel-Base Alloy," *Transactions*, Metallurgical Society, Am. Inst. Mining, Metallurgical, and Petroleum Engrs., Vol. 212, No. 4, Aug., 1958, pp. 543–548.

PREPARATION AND HIGH-TEMPERATURE PROPERTIES OF NICKEL - Al_2O_3 ALLOYS*†

By Walter S. Cremens[1] and Nicholas J. Grant[2]

Synopsis

Utilizing fine nickel powders down to 1 to 2 μ, blended with Al_2O_3 from 0.2 to 0.02 μ, extrusions were made to produce alloys containing up to 45 volume per cent of Al_2O_3. These products have been tested in tension at room temperature and in creep rupture at 1300 to 1800 F. The interparticle spacing of the Al_2O_3 in the alloy is related to the measured mechanical properties.

Ever since the disclosure in 1951 by Irmann (**1**)[3] that a finely dispersed aluminum oxide in pure aluminum gave unusual high temperature strength and high temperature structural stability, there has been a keen interest in applying the dispersion hardening principles learned from this alloy to more refractory, higher melting temperature metals and alloys. As a result there have been a number of investigations of the SAP type materials by Gregory and Grant (**2**), Lyle (**3**), and Lenel *et al* (**4,5**). In addition, Zwilsky and Grant (**6**) recently reported on the high-temperature creep rupture properties of $Cu\text{-}SiO_2$ and $Cu\text{-}Al_2O_3$ alloys which showed many characteristics similar to those exhibited by the SAP type alloys, namely, superior high-temperature creep rupture properties and structural stability at high temperatures. Similarly, interesting high-temperature properties have been shown by Bruckart and Jaffe (**7**) for molybdenum when strengthened with small additions of a variety of refractory metal oxides.

Work reported to date appears to suggest the following characteristics to be important:

1. An average interparticle spacing of less than 1 μ to achieve maximum strengthening;
2. A stable refractory particle as the means of achieving the dispersion hardening, preferentially by means of an oxide so that solubilities are either nil or exceedingly limited; and
3. Extrusion of the alloy from its powder compact form in order to achieve a high rate of deformation so that energy may be stored in the structure.

For the current work, nickel was chosen as the matrix metal because of its high melting point, its reasonable oxidation resistance in the pure form, and its availability in powder form in the size range near 1 or 2 μ. Aluminum oxide was chosen because of its high-temperature stability, its availability in reproducible fine particle form down to 0.02 μ, and because of its high melting temperature.

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† Based on a thesis submitted in partial fulfillment of the requirements for the Sc.D. degree, Department of Metallurgy, Massachusetts Institute of Technology, Cambridge, Mass.

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[3] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 729.

TABLE I.—RAW MATERIALS.

Nickel Powders	Composition, weight, per cent			Nominal Average Particle Size, μ
	Iron	Sulfur	Insoluble Precipitate	
Inco Carbonyl	0.083	0.0073	0.987	8 to 12
PR1-2	0.27	0.070	[a]	clusters 10
	0.032	0.037	0.299[b]	particles 1 to 2
PR1-2M	(same as above but micronized)			1 to 2
Inco Grade B	0.000	0.0057	0.000	2 to 3
Raney Catalyst	(2.4 per cent aluminum in prepared powder)			0.13[c]

Aluminum Oxide Powders	Diameter[d] for Uniform Spheres, μ	Specific Surface Area, sq m per g	Surface-to-Volume Ratio, cm^{-1}	Nominal Specific Gravity, g per cu cm
Alcoa (alpha)	0.22	6.97	27.6×10^4	3.96
Linde A (alpha)	0.26	5.94	23.5	3.96
Linde B (gamma)	0.05	35.0	121.5	3.47
Alon CI (gamma)	0.033	51.13	176.0	3.3 to 3.6
Alon CII (gamma)	0.018	99 to 106	334.0	3.2 to 3.3

[a] Supplier's analysis.

[b] Analysis, Massachusetts Institute of Technology.

[c] Diameter of uniform spheres corresponding to specific surface of 5.2 sq m per g.

[d] Diameter of uniform sphere corresponding to specific surface.

EXPERIMENTAL PROCEDURE

Raw Materials:

Table I lists the grades of nickel powders available to the program as well as the grades of aluminum oxide powders used in making the alloys. The PR1-2 nickel is a precipitated powder made by the Sherritt-Gordon Mines, Ltd. (8). The precipitated particles consist of weakly bonded clusters which may measure up to 10 μ in the aggregate. By using an air-blast vortex mill, the "Micronizer," it is possible to break up these clusters to produce a powder made up of discrete particles of about 1 to 2 μ. This latter micronized grade is referred to as the PR1-2 M product.

Raney catalyst, a patented powder used as a hydrogenation catalyst, was received in the form of 50 per cent nickel - 50 per cent aluminum powder and was processed according to the method of Covert and Adkins (9), essentially digestion in a sodium hydroxide solution. The resultant extremely fine powder is pyrophoric and must be stored under a liquid—in this instance alcohol. X-ray diffraction of the lattice spacing was made and compared to work of Bradley and Taylor (10). The results indicate a residual aluminum content of about 2.4 weight per cent.

The Raney nickel powder and the aluminum oxide powders were all checked to determine specific surface values according to the method of Brunauer, Emmett, and Teller (BET) (11), and the resultant values are listed in Table I.

Preparation of Powder Compacts:

Early in the program, the nickel powders and the aluminum oxide were blended by means of a ball milling operation in a ferritic stainless steel mill with ferritic stainless steel balls. The ball milling was not intended as a comminution; in fact, it was more likely that with ductile nickel powders there was a tendency to form coarser agglomerates due to welding together of nickel particles. Mixtures were ball milled for periods of 8 to 36 hr. Extrusions for alloys 14 to 29 were prepared by blending the powders by ball milling.

Subsequently a high-speed Waring Blendor was used to mix the powders. After many trials using simple tumbling, tumbling with baffles, ball milling dry and with laurel alcohol, etc., it became apparent that dry ball milling for sufficiently long times produced dispersions superior to wet methods and that blending dry in the Waring Blendor produced blends which were much more homogeneous than from the ball milling operation. Extrusions 30, 34, and 35 were made using the Waring Blendor technique. A mixing time of $\frac{1}{2}$ hr was found to give results superior to those found after many hours of ball milling.

TABLE II.—IDENTIFICATION OF EXTRUSIONS—WITH DENSITIES AND INTERPARTICLE SPACINGS.

Alloy	Volume Per Cent Oxide	Inter-particle Spacing, μ	Reciprocal of Spacing, $\mu-1$	Measured Density, g per cu cm	Per Cent of Theoretical Density	Oxide Powder Used	Nickel Powder Used
No. 14	10	5.7	0.175	8.33	99.2	Alcoa	Inco Carb.
No. 15	14.6	4.3	0.232	8.04	98.4	Alcoa	Inco Carb.
No. 16	5	9.2	0.109	8.60	99.5	Alcoa	Inco Carb.
No. 17 (pure nickel)	0	∞	0	8.90	100.0	.....	Inco Carb.
No. 18	9.9	6.8	0.147	8.45	100.5	Linde B	PR1-2
No. 19	12.1	7.5	0.133	8.18	98.6	Linde A	PR1-2
No. 20	24.7	3.9	0.256	7.49	97.5	Alcoa	PR1-2
No. 21	45.2	2.3	0.435	5.80	90.2	Alcoa	PR1-2
No. 22	10.8	6.6	0.152	8.29	99.0	Alon CI	PR1-2M
No. 23	10.1	3.8	0.262	8.35	99.4	Linde B	PR1-2M
No. 24	3.5	5.1	0.031	8.76	100.2	Alon CI	PR1-2M
No. 25	17.9	3.8	0.264	7.91	98.7	Alon CI	PR1-2M
No. 26	20.5	3.3	0.303	7.69	97.8	Alon CI	Inco B
No. 27	19.4	3.3	0.303	7.83	98.8	Linde B	PR1-2M
No. 28	20.2	3.7	0.270	7.81	99.0	Linde B	Inco B
No. 29	8.3	3.1	0.322	8.46	99.6	Alon CI	Inco B
No. 30	3.2	4.8	0.208	8.72	99.7	Alon CII	Inco B-2
No. 34	7.9	2.3	0.435	8.34	98.0	Alon CI	Inco B-2
No. 34 CW	(see values for 34)		...	8.52	100.1	Alon CI	Inco B-2
No. 35	15.2	2.1	0.476	8.02	98.4	Alon CI	Inco B-2
No. R-1	21.6	2.5	0.400	7.52	96.2	Alon CII	Raney

After the blending operation, the mixtures were reduced in dry hydrogen at 645 F for a period of three or more hours. After this reduction, the powders were stored in tightly sealed containers in preparation for making the actual powder compacts.

The compacts were prepared by tamping layers of the mixed powder into a pressing canister. The canister consisted of a brass tube about 2 in. in diameter, containing holes drilled around the entire periphery. A rubber sleeve was fitted inside the canister and was rubber stoppered at one end. Layers of the powder were tamped progressively until a powder compact approximately 3.5 in. long was formed.

This assembly was then placed in a vacuum chamber which was pumped down to 1 mm of mercury pressure, at which time the open end of the canister was stoppered.

This canister assembly was then placed in a hydraulic chamber to which a hydrostatic pressure of 75,000 psi was applied.

The cold-pressed powder compact was then sintered in dry hydrogen at a temperature of 2280 to 2370 F for a period of about 50 hr. Compact 35 was sintered at 1730 F, and compact 34 was extruded without any prior sintering operation. After sintering, the compact was ground to a right cylinder usually having the dimensions of about 1½ in. in diameter by 2 in. long.

The sintered compact was canned in a mild steel jacket in preparation for sheath extrusion. The sintered compact was dropped into the steel jacket, a plug was welded into place to seal the jacket except for the tube which was used to evacuate the can prior to extrusion. After evacuation the tube was sealed off.

The canned slug was then heated to 1950 F and extruded on a 300-ton press. The high-speed single pass reduction was calculated to give a final diameter of the nickel-alumina rod of about 0.30 in. This yielded a reduction of cross-sectional area of about 25 to 1, and permitted machining of test bars with 0.25 in. threaded ends and a 0.16 in. diameter by 1 in. long gage section. These compacts yielded about 3 ft of useful alloy for test purposes.

Exceptions to the above procedure were made for extrusions 30, 34, and 35. These were extruded on a 1000-ton capacity press and were canned in a thicker steel jacket. Also, extrusions Nos. 34 and 35 were made at a temperature of 2350 F.

Metallographic Examination:

A section from the center of each extruded rod was taken to provide transverse and longitudinal views of the structure. Specimens were mounted in bakelite and taken through the usual grinding belts and polishing papers, through No. 00 emery paper. Polishing was continued with Smit's No. 4 diamond paste on a broadcloth wheel and completed with No. 7 or No. 8 diamond paste on a velvet wheel. This procedure resulted in a minimum of tearing out of the alumina particles from the matrix. No etching technique was found which would yield an intelligible structure in the matrix while preserving the oxide inclusions. Consequently examination was made of the as-polished surface.

A Hurlbut counter was used to obtain average interparticle spacing for each alloy. Counting was carried out at a magnification of 1300×. Two perpendicular traverses were made on each specimen to give a total distance covered of 10^4 μ (1 cm) minimum. Results are given in Table II.

Creep-Rupture and Tension Testing:

All compacts were tested in the as-extruded condition with the exception of extrusion 34 which was tested also in the cold-worked condition. Stress rupture testing was carried out primarily at 1500 F since this was the highest temperature one could normally use for pure nickel without encountering excessive oxidation. This is also the temperature at which there is the greatest amount of data permitting comparison of the strength properties of these alloys with conventional alloys. A number of tests were run at 1300 and 1800 F to see what the temperature-strength characteristics of these alloys might be. All stress-rupture tests were constant load tests. Temperature control and other conditions met ASTM specifications. Test results are summarized in Table III.

Room-temperature tension tests were made on each of these extrusions to provide yield and tensile strength, elonga-

TABLE III.—STRESS-RUPTURE AND CREEP RESULTS AT 1500 F.

Alloy	Stress, psi	Rupture Life, hr	Minimum Creep Rate, per cent per hr	Reduction of Area, per cent	Elongation, per cent
No. 14	7 000	8.81	0.31	0	1.2
	5 000	148.0	0.024	0	5.8
No. 15	7 000	0.5	8.48	. . .	4.9
	5 000	1.7	2.1	0	4.5
No. 16	7 000	2.7	1.33	1.5	2.2
	5 000	118.0	0.017	1.5	3.4
No. 17 (pure nickel)	5 500	0.12	. . .	23.0	. . .
	5 000	0.2	45.0	90.0	31.0
	4 570	0.73	3.9	6.0	13.0
	2 980	8.97	. . .	5.0	8.9
No. 18	7 000	5.2	. . .	5.1	2.3
	5 000	106.0	0.032	. . .	5.3
No. 19	7 000	1.2	2.1	2.5	2.6
	5 000	5.9	0.4	1.0	2.4
No. 20	7 000	5.5	. . .	. . .	1.2
	5 000	30.9	. . .	. . .	5.0
No. 22	7 000	1.5	1.83	2.8	2.9
	7 000	3.1	1.1	2.5	5.0
	5 000	11.0	. . .	2.0	4.5
	5 000	8.1	0.19	1.3	3.6
No. 23	9 000	21.6	. . .	1.3	. . .
	8 000	22.8	. . .	2.6	. . .
	7 000	245.0	0.02	0.0	4.6
	7 000	59.1	. . .	0	. . .
	5 500	1003.6	. . .	0	. . .
	5 000	188.0	0.0001	. . .	4.7
No. 25	9 000	3.6	. . .	2.5	. . .
	7 000	17.8	0.250	0	5.3
	5 000	433.0	0.006	0	5.6
No. 26	9 000	5.6	. . .	1.9	. . .
	7 000	24.0	. . .	1.3	2.0
	6 000	128.8	. . .	0	. . .
No. 27	9 000	1.4	. . .	3.7	. . .
	7 000	48.9	. . .	1.9	. . .
	7 000	27.4	. . .	1.2	. . .
	5 500	378.1	. . .	0	. . .
No. 28	9 000	4.2	. . .	3.2	3.9
	7 000	16.5	. . .	1.9	2.4
	7 000	16.0	0.37	1.3	5.6
	6 000	607.0	0.008	0	6.5
	5 000	525.0	0.0023	. . .	4.3
No. 29	9 000	3.5	. . .	2.0	. . .
	7 000	58.8	0.065	6.4	4.2
	6 000	274.0	0.0057	0	2.0
	5 500	1664.0	0.00033	0.6	5.2
	5 000	373.0	. . .	0	2.4
No. 30	7 000	0.31	9.9	4.3	3.1
	5 000	0.73	3.4	5.0	4.5
No. 34	14 000	3.8	. . .	2.5	0.8
	11 000	1.9	. . .	6.0	. . .
	9 000	4.02	. . .	0	. . .
	7 000	213.0	0.0034	. . .	. . .
No. 34 CW	14 000	1.7	. . .	1.0	. . .
	12 500	23.7	0.135	3.7	3.1
	11 000	132.1	0.027	0.5	3.4
No. 35	14 000	2.57	1.3	2.5	3.7
	11 000	27.1	0.067	0	2.8
	9 000	104.4	0.011	0	1.1
	9 000	31.3	0.053	0	2.6
	7 000	218.0	0.0018	. . .	. . .
No. R-1	9 000	0.79	. . .	0	2.5
	7 000	0.55	3.5	0	2.6
	7 000	0.13	. . .	0	. . .
	5 000	36.8	0.029	0	2.3

TABLE III.—*Concluded.*

Alloy	Stress, psi	Rupture Life, hr	Minimum Creep Rate, per cent per hr	Reduction of Area, per cent	Elongation, per cent
		1300 F			
No. 17 (pure nickel)	6 000	3.6	...	9.8	...
No. 25	15 000	16.1	...	3.1	...
	13 000	44.0	...	1.0	...
	11 000	161.7	...	...	...
No. 35	18 000	18.7	...	3.5	...
	15 000	385.0	...	...	...
		1350 F			
No. 14	10 000	26.5	...	2.7	2.4
No. 15	10 000	1.4	...	...	2.0
No. 16	10 000	11.4	...	...	1.2
No. 17 (pure nickel)	5 500	2.45	1.0	9.7	15.0
	4 000	14.5	...	5.4	7.0
	3 030	68.7	0.06	5.9	18.0
		1800 F			
No. 17 (pure nickel)	3 029	0.53	...	6.2	...
No. 25	4 500	2.02	...	0	...
No. 26	5 000	0.95	...	0	...
No. 28	5 000	0.15	44.0	3.1	5.6
	3 000	0.15	44.0	3.1	5.6
	3 040	360.0	...	...	...
No. 29	8 022	0.13	...	3.7	...
	2 890	118.1	0.013	6.2	4.1
No. 35	7 000	1.0	1.6	0	1.6
	5 000	5.4	0.13	0	2.8

TABLE IV.—ROOM TEMPERATURE TENSILE PROPERTIES.

Alloy	Ultimate Tensile Strength, psi	Yield Strength 0.2 per cent Offset, psi	Elongation,[b] per cent	Reduction of Area, per cent	Rockwell Hardness, A Scale
No. 14	61 900	36 400	20.0	17.0	53.0
No. 15	59 100	43 500	11.0	7.2	50.0
No. 16	59 800	48 000	27.0	39.0	46.0
No. 17 (pure nickel)	65 000	50 000	20.0	74.0	27.0
No. 18	65 300	39 800	12.5	11.2	57.0
No. 19	53 700	33 900	12.5	9.2	52.0
No. 20	82 300	65 200	4.2	3.5	61.0
No. 22	53 600	33 500	12.0	9.8	52.0
No. 23	72 700	40 500	13.0	28.0	44.0
No. 25	75 500	49 300	6.7	3.7	54.0
No. 27	75 500	55 000	9.3	12.5	53.0
No. 28	80 200	53 200	4.0	3.8	55.0
No. 29	52 500	34 500	27.0	38.0	44.0
No. 30	56 300	18 800	44.0	43.0	...
No. 34	54 200	25 900	24.0	31.0	45.0
No. 34 CW	63 000	60 000	10.7	18.2	54.0
No. 35	61 100	...	8.0	3.7	...
No. R-1	71 500	58 400	5.3	2.5	...
No. A—Ni[a]	50 000–80 000	10 000–30 000	35.0–50.0	50.0–70.0	31.0–42.0

[a] Annealed A Nickel (99.4 nickel + cobalt) values from "ASM Metals Handbook," 1948 Edition, Am. Soc. Metals, p. 1046.

[b] 1-in. gage length.

tion, and reduction of area values. These results are summarized in Table IV.

TEST RESULTS

1500 F Creep-Rupture Results:

The principal strength criterion chosen for these materials was rupture life at 1500 F (Fig. 1). For most of the materials only two or three datum points were obtained. These were plotted separately for individual materials, and the most representative straight line was transposed to Fig. 1. Straight lines on a semilog plot were found to give a fair representation of the data for each alloy.

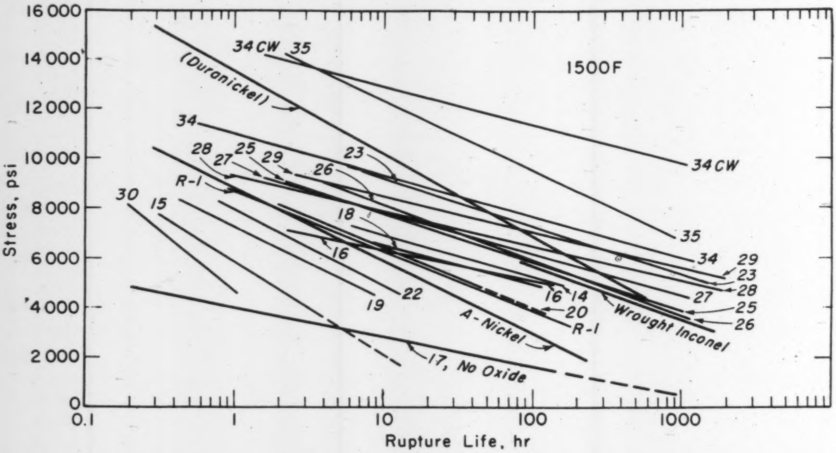


FIG. 1.—Stress *versus* Log Rupture Life at 1500 F for Ni-Al_2O_3 Alloys, Commercial Nickel, Inconel and Duranickel.

Included for comparison are four alloys containing no oxide additions: No. 17, a nickel powder process blank; wrought A-nickel, commercially pure nickel; wrought Duranickel (in the annealed condition), an alloy containing 4.3 per cent aluminum; and Inconel (12).

The topmost curve, 34 CW, represents tests made on extrusion 34 in the cold-worked condition. Part of the extrusion was swaged at room temperature, still in its steel extrusion jacket, to an area reduction of 37 per cent. The height of curve 34 CW above that for as-extruded 34 shows the strength increase due to cold working and further densification. All other materials were tested only in the as-extruded condition.

Comparing the 34 CW alloy to the reference alloy No. 17, a sevenfold increase in the stress for 100-hr rupture life value was obtained with this alloy containing 7.9 volume per cent of 0.03 μ aluminum oxide. Sixfold increases were obtained with several other alloys.

The rupture life properties in Fig. 1 are correlated with the structures of the alloys in the next two figures. Some scatter is to be expected, as shown in Fig. 2, because of differences in segregation from alloy to alloy and because of density differences over a fairly wide scale. Figure 2 is a plot of stress for 10-hr rupture life *versus* the reciprocal of the average interparticle spacing. The top scale conveniently shows the interparticle spacing directly. The boxed datum points represent alloys which had less than 99 per cent of theoretical density and which were accordingly weighted less heavily than the more dense alloy values in drawing the curve. Similar curves were determined for the 100-hr and 1000-hr rupture life stress values, and the summary of curves is shown in Fig. 3.

By comparison, plots of stress for a given rupture life *versus* volume per cent oxide did not show a consistent pattern at all.

Because of a limited number of test specimens and relatively short-time creep rupture tests, it was impossible to obtain many complete creep curves; however, based on values available, Fig. 4 shows the approximate minimum creep rates for a number of the alloys. Compared to pure nickel (alloy No. 17), alloys Nos. 23 and 29 show a decrease in creep rate at 5000 psi and 1500 F by a factor of about 10^5.

Elongation and reduction of area values are listed in Table III. The reduction of area values are somewhat questionable because of significant oxidation during test, especially in longer time tests.

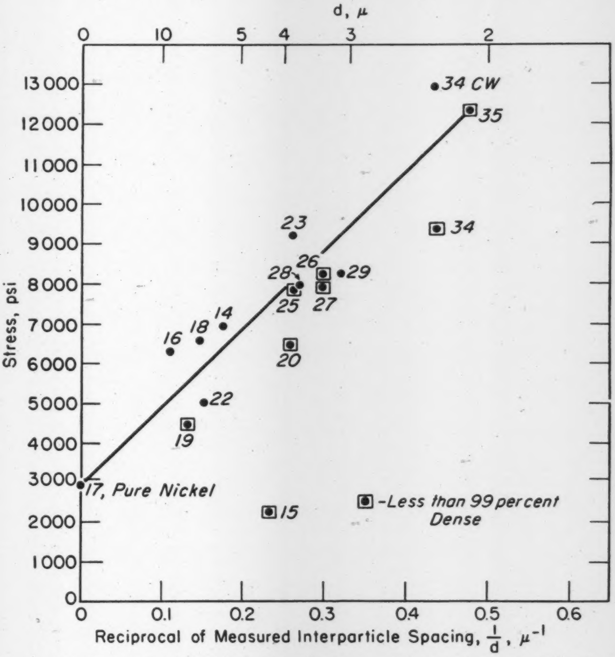


Fig. 2.—Stress for 10-hr Rupture Life at 1500 F *versus* Reciprocal of Interparticle Spacing.

The 1300 and 1800 F Creep Rupture Tests:

The creep rupture tests which were run on a limited number of alloys at 1300, 1350, and 1800 F are listed in Table III and yielded values of stress for a 10-hr life. These results are summarized in Fig. 5 as a function of the test temperature and are compared to listed values for the commercial alloy, Inconel, and pure nickel. The importance of full density is apparent, especially at the higher temperatures, since alloys Nos. 28 and 29 show much more flat slopes than do alloys Nos. 25 and 35.

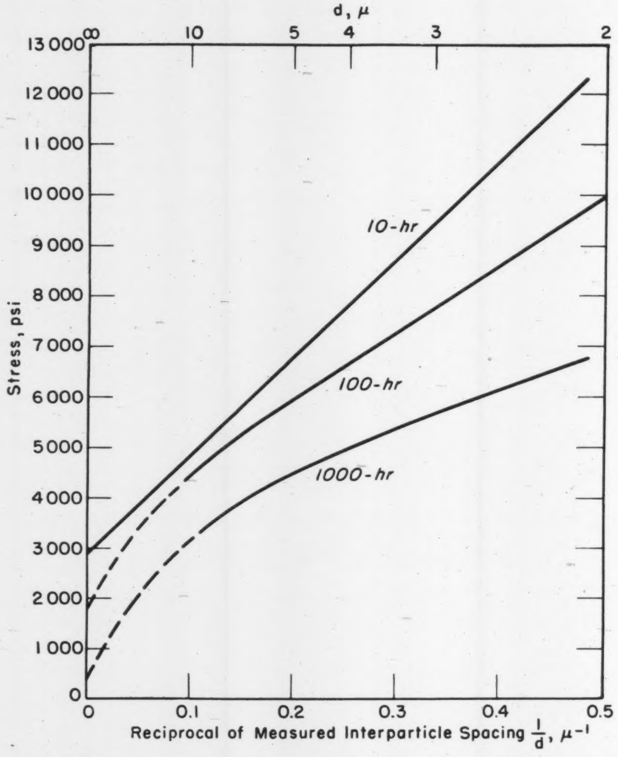


FIG. 3.—Stress for Rupture Time Values of 10, 100 and 1000 hr at 1500 F *versus* Reciprocal of Interparticle Spacing for Ni-Al_2O_3 Alloys.

Room Temperature Tensile Data:

Ultimate tensile and yield strength values are plotted in Fig. 6 (*a*) while elongation and reduction of area are plotted in Fig. 6 (*b*), both against volume per cent of oxide. The volume per cent oxide values gave a better relationship in this instance than did interparticle spacing. The effect of oxide additions on the room temperature tensile or yield strength is relatively small compared to the effect on high-temperature strength. In contrast to this, the effect of volume per cent oxide on ductility is large. Note here, too, the apparently important effect of density on the ductility values (in contrast to the effect on strength). Alloys containing less than 10 volume per cent of oxide show relatively high elongation values.

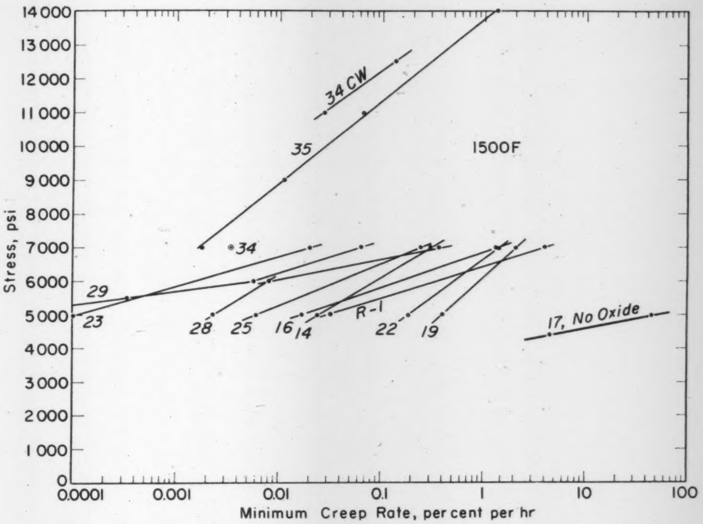


Fig. 4.—Stress *versus* Log Minimum amp Rate at 1500 F for Ni-Al_2O_3 Alloys.

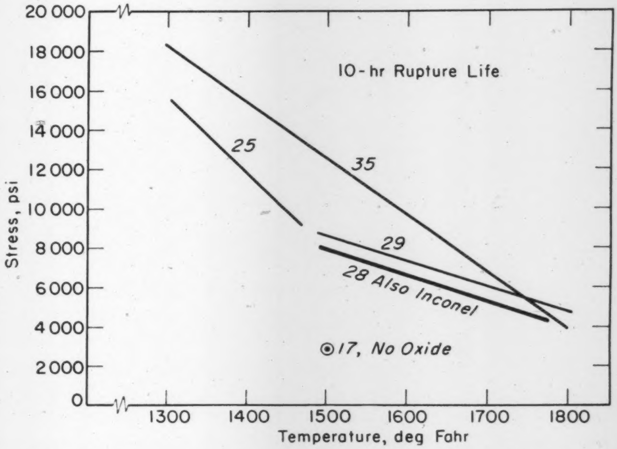
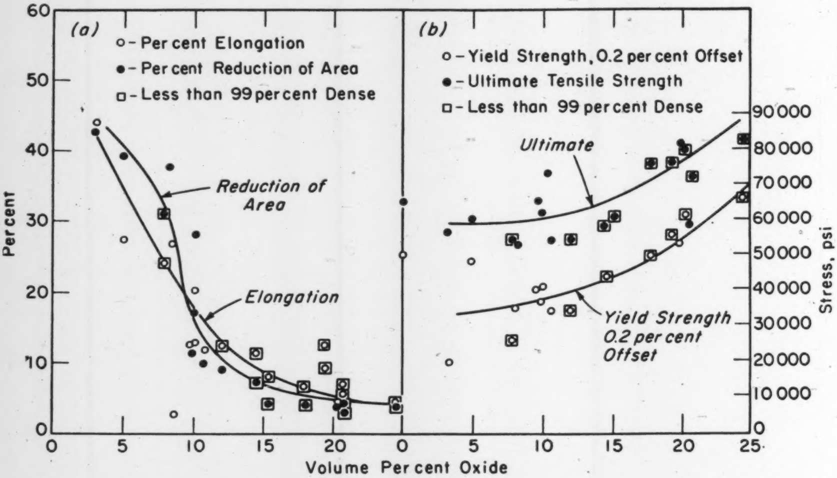


Fig. 5.—Stress for 10-hr Rupture Life *versus* Temperature Compared to Pure Nickel and Inconel.

Metallographic Observations:

Early tests indicated the difficulty of reproducing reliable, accurate structures in polished specimens. Electron micrographs showed oxide particles up to 50 times larger in alloy No. 18 than the 0.05 μ Linde B alumina originally added. Furthermore, although line width measurements were not treated quantitatively, measurements on extracted particles from extrusion No. 18 showed alpha-alumina (gamma was added), the particles of which were significantly coarsened. It clearly appears that clusters of fine gamma alumina had sintered into coarse alpha particles. Accordingly it appeared adequate to use a light microscope with a Hurlbut counter to measure particle spacing at 1300×.

Typical transverse and longitudinal sections are shown in Figs. 7 and 8. Figure 7 is of alloy No. 29, which contains 8.3 volume per cent Al_2O_3 with an interparticle spacing of 3.1 μ. Figure 8 is of alloy No. 35 with 15.2 volume per cent of Al_2O_3 with an interparticle spacing of 2.1 μ. The amount of stringering of the oxide in these two alloys is relatively small.



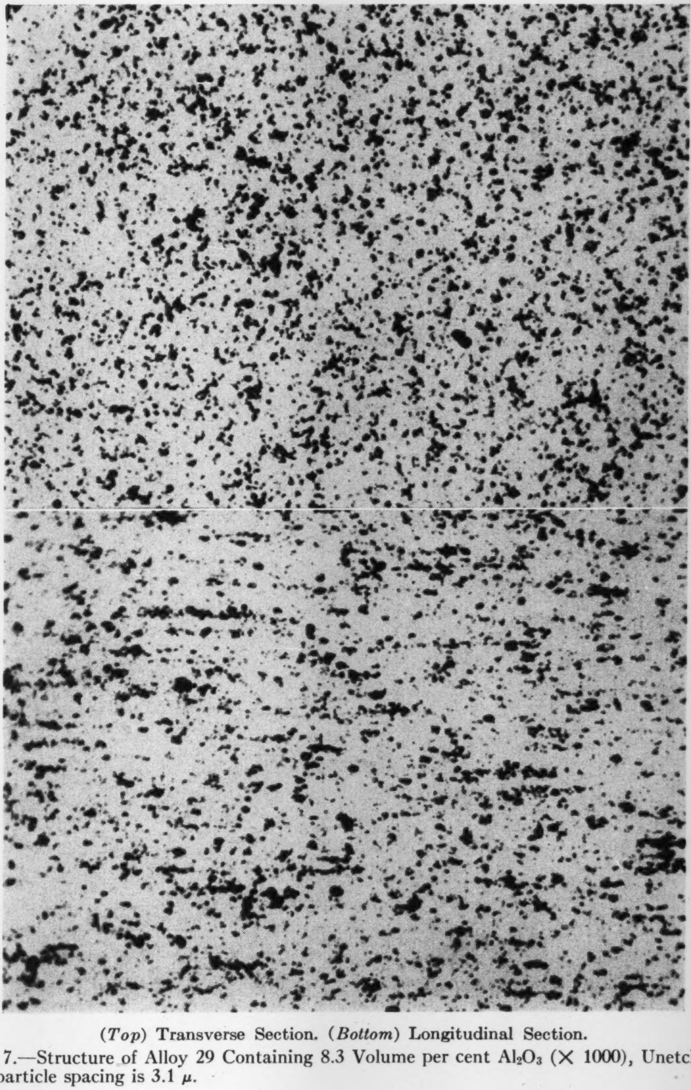
(*a*) Elongation and Reduction of Area. (*b*) Ultimate Tensile Strength and Yield Strength.

FIG. 6.—Room Temperature Tensile Properties *versus* Volume per cent Oxide.

DISCUSSION

Variations in Properties.—Table I shows that the PRl-2 nickel powder had 0.070 per cent sulfur (our check analysis gave 0.037). The other raw materials were generally lower in sulfur, but most were considerably above the optimum maximum of 0.005 per cent desired for nickel alloys. Fortunately, most of the extrusions showed less than 0.020 per cent and many showed below 0.005, except alloy No. 24 which had 0.015 and alloy R-1 which had 0.017 per cent. It would appear that the hydrogen sintering operation is instrumental in lowering the sulfur content of the nickel powder while it is in the low-density compact form.

The accuracy of the theoretical density values listed in Table II is believed to be ±1 per cent. For this reason the points in Figs. 2 and 6 for alloys with less than 99 per cent density are boxed. It was observed for the fabrication techniques used in this study that densities greater than 99 per cent are usual for alloys containing less than about 10 volume per cent of Al_2O_3 and generally are less for alloys with greater than 10 volume per cent oxide (see Table II). It

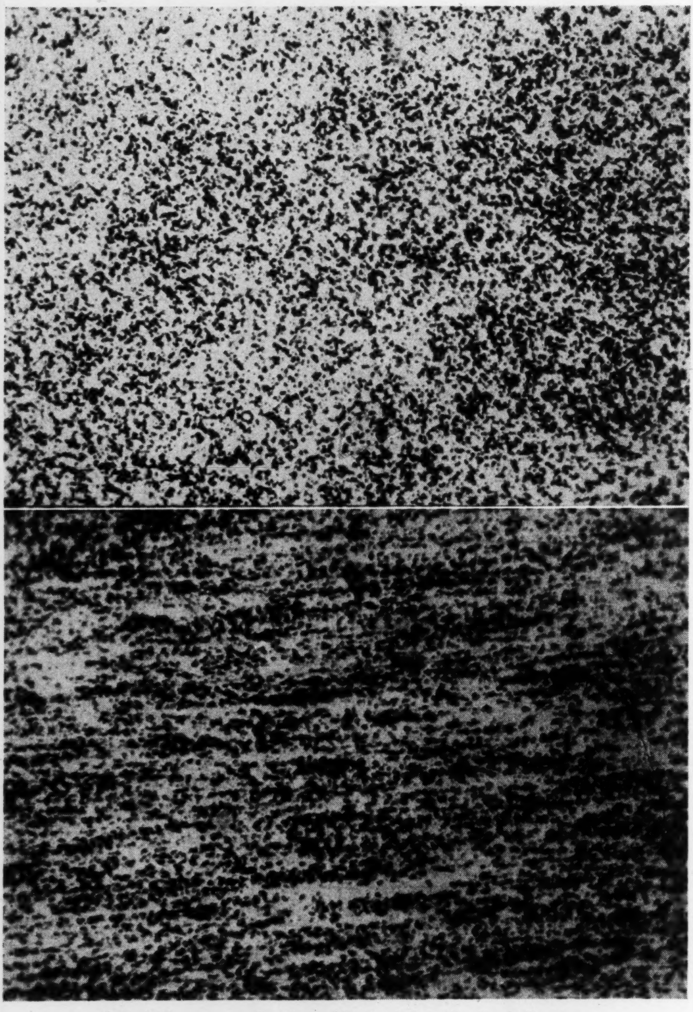


(*Top*) Transverse Section. (*Bottom*) Longitudinal Section.

FIG. 7.—Structure of Alloy 29 Containing 8.3 Volume per cent Al_2O_3 (× 1000), Unetched. Interparticle spacing is 3.1 μ.

is presumed that the large improvement in the stress rupture properties of alloy 34 CW was due in substantial part to improvement in density with cold work.

Strength Considerations.—While large increases in rupture life and creep resistance are evident from Figs. 1 and 4, it is Figs. 2 and 3 that are of particular interest. Recalling that the interparticle spacing of the almost ideal structure of



(*Top*) Transverse Section. (*Bottom*) Longitudinal Section.

FIG. 8.—Structure of Alloy 35 Containing 15.2 Volume per cent Al_2O_3 (× 1000), Unetched. Interparticle spacing is 2.1 μ.

SAP was about 0.35 μ (2), extrapolation of the curves of Figs. 2 and 3 clearly indicates that still further strength improvements are to be anticipated if finer interparticle spacings can be achieved by mechanical blending of ultra fine metallic and oxide powders.

Interparticle Spacing.—In the present work and elsewhere (2,13,14), it has been shown that the average interparticle

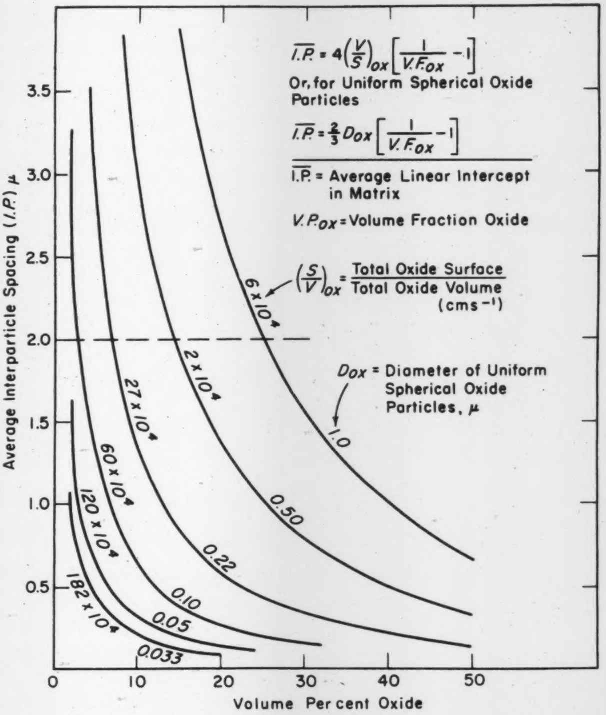


FIG. 9.—Calculated Average Interparticle Spacing *versus* Oxide Content.

spacing of the second phase is the structural parameter of prime importance for creep strength of dispersion strengthened materials.

Assuming an alloy consisting of isolated oxide particles in the matrix phase, the alloy to be made by mixing of assumed uniform spheres of oxide and metal, what is the relation between the amount and particle size of oxide and the average interparticle spacing $\overline{I.P.}$?

The answer, derived from general spatial relations given by Smith and Guttman (**15**) is:

$$\overline{I.P.} = 4\left(\frac{V}{S}\right)_{ox}\left[\frac{1}{V.F._{ox}} - 1\right] \quad \ldots (1)$$

where:

$\overline{I.P.}$ = arithmetical average of interparticle spacings determined as the mean value of the lengths of intercepts made on a random line by oxide-matrix interfaces,

$\left(\frac{V}{S}\right)_{ox}$ = ratio of total volume of oxide to total oxide surface, and

$V.F._{ox}$ = volume fraction of oxide,

The term $\left(\frac{V}{S}\right)_{ox}$ expresses the oxide "particle size." For instance, for spherical particles of uniform size:

$$\left(\frac{V}{S}\right)_{ox} = \frac{D_{ox}}{6}$$

where D_{ox} is the particle diameter.

Using Eq 1, Fig. 9 was constructed

showing $\overline{I.P.}$ as a function of volume per cent oxide for several different oxide particle sizes. For one simple shape, namely, uniform spherical particles, diameters are noted to aid visualization, but the relation in terms of volume to surface ratio is perfectly general.

Figure 9 shows the minimum interparticle spacing which it is possible to achieve under ideal conditions with a given proportion of oxide powder of a stated size. This is the reference figure which was utilized in selecting materials to produce alloys of desired structure.

Suppose it is desired to obtain an interparticle spacing of 1 μ using less than 15-volume per cent of oxide. Figure 9 shows that an oxide powder of 0.5-μ diameter is too coarse and indicates a particle size of about 0.22 μ would be needed even with ideal conditions.

Ideal conditions concern the difference between mean and average interparticle spacings and involve two major requirements: the first is a general requirement of perfect mixing, that is, a random dispersion of individual oxide particles. The second requirement involves the metallic powder particle size, which cannot be much greater than the mean interparticle spacing desired. For instance, if the metal powder particles are uniform spheres, their diameters, D_m, cannot exceed $\frac{3}{2}$ of the mean interparticle spacing desired.

If either condition is violated, the discrepancy between mean and average interparticle spacing becomes large. The average interparticle spacing, *I.P.*, is a property of the body, rigorously fixed by Eq 1. In a segregated body, however, having for example clumps of oxide particles with oxide-free areas between, the average spacing is taken among many very short intercepts within the clumps and a few large intercepts between clumps. The average interparticle spacing in such a case, while it remains a property of the body as a whole, no longer approximates the mean value and is no longer representative of the structure as regards its mechanical behavior.

There should be no confusion, however, between $\overline{I.P.}$ and the experimental values, *d*, of Table II. The quantity $\overline{I.P.}$ is mathematical and ideal. The *d*-values are experimental and were measured on nonrandom sections with an optical method of barely adequate resolution. What $\overline{I.P.}$ does show is the limiting minimum value which *d* approaches as both the experimental conditions of observation and the degree of mixing improve.

Any of several sizes of oxide powder may be used. For instance, 3 volume per cent of 0.1-μ oxide powder, 14 volume per cent of 0.5-μ powder, or 25 volume per cent of 1.0-μ powder will give an average interparticle spacing, $\overline{I.P.}$, of 2 μ (which under the ideal conditions assumed will not differ greatly from the mean value).

If more than the necessary amount of oxide is added, corresponding to any point on a curve below the line $\overline{I.P.} = 2\ \mu$, a condition of "overloading" occurs. Segregation must take place, and the values for average and mean interparticle spacing diverge. The practical consequence in a mixture pressed and extruded is stringer formation with anisotropic properties resulting. An alternative phenomenon to simple stringer formation observed was oxide-to-oxide sintering by which clusters of oxide combine to give a coarser effective oxide particle size than that in the raw materials.

In such a case, the volume-to-surface value required for Eq 1 cannot be measured on the raw oxide powder but must be determined *in situ*.

When mixing techniques do not give perfect dispersion, it may be necessary to overload the mixture, accepting some clustering in order to decrease the mean value.

The belief is widely held that for metal-ceramic combinations to maintain the ductility characteristics of metals, the ceramic component must be held below about 15 or at most 20 volume per cent. The room temperature results in the present work would seem to bear out this idea. If it is accepted that volume per cent oxide should be held below some arbitrary value and if mean interparticle spacings of the order of 1 μ or smaller are desired (see Fig. 3), then Fig. 9 indicates the range of metal and oxide particle sizes which must be used.

SUMMARY AND CONCLUSIONS

1. The method of mechanical mixing of powders to attain dispersion strengthened alloys of the SAP type has been firmly established. It has been shown that improved properties can be achieved in the absence of the ideal distribution of oxide on fine metallic powders, typical of the SAP type product.

2. The importance of the interparticle spacing as the definitive parameter relating dispersion-structure to creep resistance has been demonstrated again. An approximately linear relation was found between stress-for-rupture in a fixed time and the reciprocal of the interparticle spacing.

3. A topological relation (Fig. 9) was developed to relate average interparticle spacing in the matrix to the proportion and specific surface ("particle size") of the dispersed phase.

4. Stress-rupture results observed for the strongest extrusions are comparable to those for more complex commercial alloys.

5. High values for room temperature ductility were found for extrusions containing less than 10 volume per cent of oxide. At 1500 F the elongation was generally in the range of 2 to 4 per cent for all of the materials.

6. Room temperature strength properties were only slightly affected by the oxide additions as compared to their effect at elevated temperatures.

7. Extrapolation of the results in hand points to the possibility of achieving "super-alloy" rupture strength with a still finer dispersion of aluminum oxide in an otherwise unalloyed nickel matrix.

Acknowledgments:

The authors wish to thank Gloria Johnson for her able assistance in the metallographic portions of this work. Grateful acknowledgment is made of the materials and services received from the firms and laboratories mentioned in the text. This program was initiated under contract from Wright Air Development Center and was completed under the sponsorship of The International Nickel Company.

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DISCUSSION

MR. CLAUS G. GOETZEL[1] (*presented in written form*).—The authors are to be complimented on this most interesting investigation. Of particular value appears to be their attempt at correlating, by experiment and mathematical analyses, the interparticle spacing of the second phase with the creep strength and stress rupture life of the nickel-aluminum oxide alloys. The conclusions which the authors have drawn relative to the ideal minimum spacing of the second phase particles may conceivably be of considerable significance in the development of other high-temperature-resistant dispersion alloy systems.

The investigation has raised two interesting questions which deserve further attention. The first concerns the results obtained with the extrusion 34 CW that was subsequently cold worked. Are the superior results—especially the flatter slope of the stress rupture curve at 1500 F in Fig. 1—solely to be attributed to an increased density, that is, the theoretical value? Or is it perhaps possible that the cold work has stored additional energy in the structure and the strained matrix is not yet sufficiently stress relieved at 1500 F? It would be interesting to know how such cold-worked extrusion would behave at higher temperatures, for example at 1800 F. If the effect should prove to be solely a function of the increased density, would not subsequent hot work, such as swaging, produce a similar improvement? In view of the great potential which such a subsequent working procedure holds in store from a viewpoint of industrial alloy development, it would seem worthwhile if the authors would further investigate this approach, and perhaps confirm a trend with other matrix materials.

The second question I have concerns the experiment carried out with the Raney nickel powder. Off hand, this matrix material had the best chance to fulfill the requirement of still finer dispersions of aluminum oxide in a nickel matrix to achieve rupture strength comparable to that of the present super alloys, as cited by the authors in point 7 of their conclusions. Unfortunately, the results with this powder were disappointing. Is it correct to attribute this to a very high aluminum oxide content of the single extrusion reported? Would the authors care to speculate whether a lower aluminum oxide proportion, perhaps in the form of coarser particles of the Alcon CI type, would have resulted in a more homogeneous dispersion and better rupture strength? Finally, it would be interesting to know, whether the 2.4 per cent residual aluminum, converted partly or fully to the oxide by some kind of internal oxidation mechanism, would have a beneficial effect without the addition of aluminum oxide powder.

MR. N. J. GRANT (*author*).—Alloy 34 was cold worked, in which case it became 34 CW. Our reaction is that the improvement in properties was due to the improvement in density. Other alloys in this program showed lower stress rupture values when the density was 2 to 4 per cent less than 100. Hot work would have given us comparable results; we used cold work because appropriate facilities were available to us.

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[1] Senior Research Scientist, New York University, Department of Metallurgical Engineering, New York, N. Y.

As pointed out in the case of our aluminum - aluminum oxide alloys,[4] large amounts of cold work did not change our hot properties significantly (when the alloy was at full density), nor did annealing after cold work change these same properties.

We assume that comparable results would have been noted at 1800 F as at 1500 F, but unfortunately did not have sufficient bar stock for more extensive tests.

The Raney nickel tests were indeed disappointing. We ascribe the poor results to the fact that this powder product, while having very high specific volume, is rather coarse, being made up of a porous spongelike mass after leaching. Accordingly we believe that our oxide additions were poorly distributed and resulted in a segregated structure which was worse than that obtained with our regular grades of fine nickel.

Based on recent internal oxidation studies of a dilute nickel-aluminum alloy, we think that 2.4 per cent aluminum could produce significant strength improvements if properly converted to oxide.

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[4] W. S. Cremens, E. Bryan, and N. J. Grant, "Temperature and Time Stability of M-257 and S. A. P. Aluminum - Aluminum Oxide Alloys," p. 753, of this publication.

STRESS-RUPTURE PROPERTIES OF MALLEABLE IRON AT ELEVATED TEMPERATURES*

By Lauriston C. Marshall[1] and George F. Sommer[2]

Synopsis

Experimental work in progress has yielded data concerning high-temperature stress-rupture and creep properties of unalloyed malleable irons. Both ferritic and pearlitic materials have been tested for times to 2300 hr and temperatures ranging from 800 to 1200 F. Most applications are believed to fall within these ranges. Plots of log stress *versus* log rupture time as well as log stress *versus* $T(C + \log t) \times 10^{-3}$ (Larson-Miller plots) show no discontinuities in maximum test periods or at any temperature thus far. This, with careful metallographic inspection, supports the conclusion that all materials tested to date exhibit only highly ductile properties and small likelihood of transitions or structural changes. Lines and extrapolations presented are based upon least squares calculations from the actual data with statistical confidence limits. A new least squares method of computing the parameters for a Larson-Miller plot has also been derived analytically and is presented. The statistical methods applicable to a calculation of this kind and precautions to be observed in applying extrapolations are discussed. The results indicate a high level of performance to be expected from ferritic malleable irons at 800, 1000, and 1200 F, with a distinct anomalous behavior on the part of low-carbon ferritic material which exhibits unexpectedly high strength at 800 F. Data for pearlitic malleable irons thus far also indicate a high level of performance. These pearlitic malleable irons appear superior to unalloyed cast irons with which they are often compared and equal or superior in performance to a number of the alloyed materials.

Although considerable information is available on the tensile properties of malleable iron at room temperature, there are relatively few publications concerning elevated temperature properties. Notable among these are a joint ASTM-ASME publication (**1**),[3] covering tests conducted at Battelle Memorial Inst. sponsored by the War Metallurgy Division of NDRC (**2**), and unpublished data obtained at the University of Wisconsin under the sponsorship of a member of the Malleable Founders' Society (**3**). Results of these studies are limited to short-time properties in tension over a temperature range from approximately 75 to 1200 F. Such information is inadequate for designing equipment intended to operate for long intervals at elevated temperatures. Here some knowledge of the stress-rupture properties is

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* Presented at the Sixty-first Annual Meeting of the Society, June 22–27, 1958.

[1] Director of Research, Link-Belt Co., Indianapolis, Ind.

[2] Research Engineer, Link-Belt Co., Indianapolis, Ind.

[3] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 751.

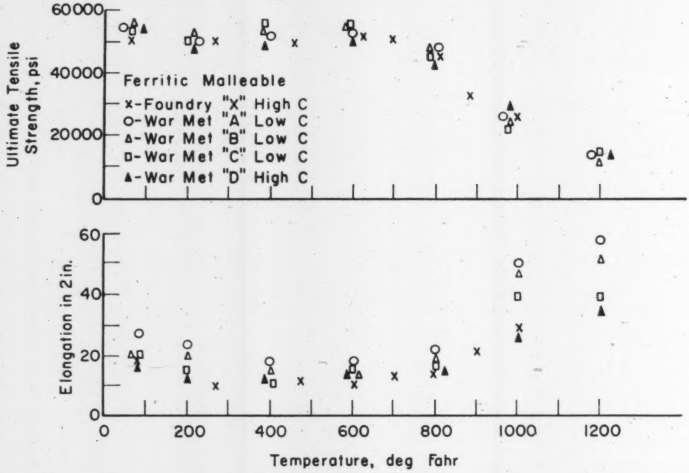

FIG. 1.—Effect of Temperature on Short-Time Tensile Properties of Ferritic Malleable Iron. Elongation values in per cent.

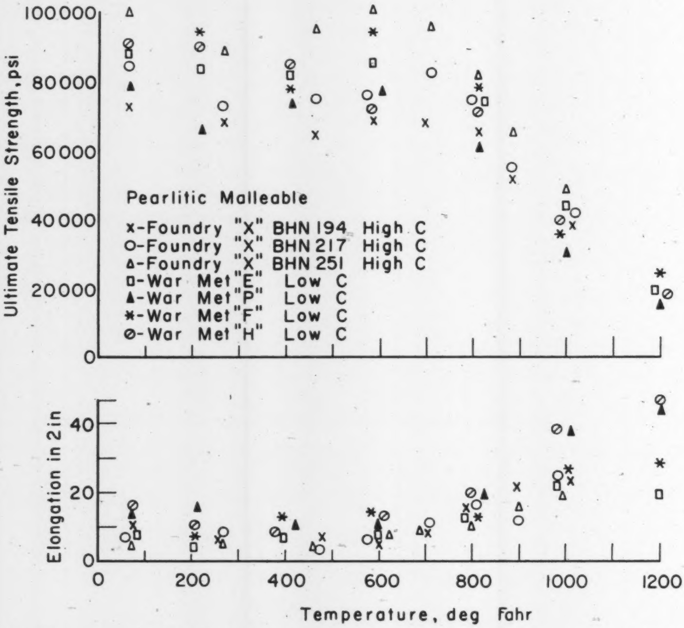

FIG. 2.—Effect of Temperature on Short-Time Tensile Properties of Pearlitic Malleable Iron. Elongation values in per cent.

essential; and, if deformation is important, observations of creep behavior are necessary. Only two publications (4,5) deal with the stress-rupture and creep properties of malleable iron. One of these studies was carried out with cupola malleable iron, which is not typical of most commercial grades of malleable iron. The other is principally concerned with the effect of alloy additions. Stress-rupture testing of malleable iron is in progress at the University of Michigan under the sponsorship of a producer of malleable iron, but results have not been published.

The uses of malleable iron, both pearlitic and ferritic, at elevated temperatures have recently been increasing. The absence of reliable creep and stress-rupture data tends to handicap the application of these materials to entirely new uses where no similar experience is available. In addition, certain types of equipment are designed around existing codes and standards, which tend to prevent full use being made of materials for which adequate design information is not available.

There is also the possibility that standard grades of pearlitic and ferritic malleable iron may be improved considerably for elevated temperature service through suitable alloying additions.

In view of this situation the Malleable Founders' Society decided upon a program designed to obtain useful data in the desired areas. This was established at Purdue University as sponsored research, employing existing stress-rupture testing facilities. The work is a companion program to fundamental research on the equilibrium thermodynamics of malleable iron constituents in the solidification range of temperatures. It is planned to obtain adequate design information to assist in applying ferritic and pearlitic malleable irons to applications requiring load-carrying ability for long periods of time at elevated temperatures. Stress-rupture and creep data have been obtained together with metallographic studies of the failed test specimens. All elevated temperature measurements reported or discussed in this paper have been obtained in air at normal pressures.

Prior to initiation of the stress-rupture tests, information then available (1,2,3) was reviewed. Figures 1 and 2 summarize data for standard and pearlitic compositions, respectively, contained in the Battelle Memorial Inst. (2) and the University of Wisconsin (3) reports. These cover short-time elevated temperature observations. It can be seen that tensile strength (upper half of Figs. 1 and 2) shows very little change with temperature until in the neighborhood of 700 F, beyond which there is a rapid decrease with increase in temperature both for standard ferritic and pearlitic malleable irons. A decrease in elongation likewise begins immediately above room temperature (lower half of Figs. 1 and 2), reaching the minimum at 400 to 600 F for both standard and pearlitic and then increasing to values as high as 60 per cent in a 2-in. gage length at 1200 F for standard ferritic malleable irons. Yield strength and reduction of area follow the same pattern as tensile strength and elongation, but are not shown in this paper because of space limitations. For the present work, an upper limit of 1200 F was chosen because of the relatively low strengths beyond this temperature. Tests were conducted at 800, 1000, and 1200 F with standard and pearlitic malleable iron supplied by five producers.

MATERIAL AND PROCEDURE

The materials were carefully selected to be representative grades of malleable iron currently in production. Test specimens of both grades of standard ferritic malleable iron, high- and low-carbon analysis were supplied from production heats by a number of foundries. Several grades of pearlitic malleable iron are likewise under test. Some data on the latter are presented in this paper. Chemical analyses are given in Table I. A division into two groups is apparent, classified as low-carbon (2.00 to 2.45 per cent carbon) and high-carbon (2.30 to 2.70 per cent carbon) (6).

Standard 0.505-in. diameter threaded bars (ASTM Specification E 21)[4] with 2-in. gage length were employed except for pearlitic malleable specimens at 800 F, where higher stresses are required. In this case, standard $\frac{3}{8}$-in. specimens with threaded ends (ASTM Specification E 8)[4] were used.

Load was applied to the stress-rupture specimen by means of weights and a nominal 10:1 lever arm. Friction in the lever was reduced by means of hardened steel knife edges and anvils. Three loading systems were available, each calibrated by means of a proving ring before tests and at suitable intervals during the program.

Three thermocouples were located at each end and in the center respectively of the gage length of each test specimen. Temperatures were measured with a precision standard cell and potentiometer combination. Specimen and furnace can be adjusted relative to one another, thus obtaining uniform temperature across a gage length within a range of 3 F. The center thermocouple on each specimen was connected to a multiple-point temperature recorder to furnish a continuous record of the test, and to permit detection of any deviation in temperature.

Time to rupture was measured by means of direct reading clocks capable of indicating 0.1 hr. These are connected through microswitches actuated to stop the clock when the specimen fails.

Elongation was measured by means of dial gages calibrated in 0.001 in. These measured movement of the upper portion of the test specimen train as indicated, permitting observation over a very wide range of extension in a manner compatible with ASTM Specifications E 139.[4] The assumption was made that elongation outside the specimen gage marks is negligible. Comparison of the dial gage readings with per cent elongation obtained from the specimen after failure confirmed this assumption and indicated that the values obtained are within 5 per cent of the true magnitude of elongation.

TABLE I.—CHEMICAL ANALYSES OF TEST SPECIMENS.

Material	Composition, per cent					
	Carbon	Silicon	Manganese	Sulfur	Phosphorus	Chromium
Ferritic (low-carbon):						
Group A-1	2.26	1.09	0.29	0.074	0.148	...
E-1	2.16	1.17	0.38	0.095	0.137	0.017
G-1	2.29	1.01	0.38	0.086	0.11	...
Ferritic (high-carbon):						
Group B-1	2.50	1.32	0.43	0.159	0.024	0.029
Pearlitic (low-carbon):						
Group E-2	2.27	1.15	0.89	0.098	0.135	0.019
G-2	2.29	1.01	0.75	0.086	0.11	...

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[4] 1958 Book of ASTM Standards, Part 3.

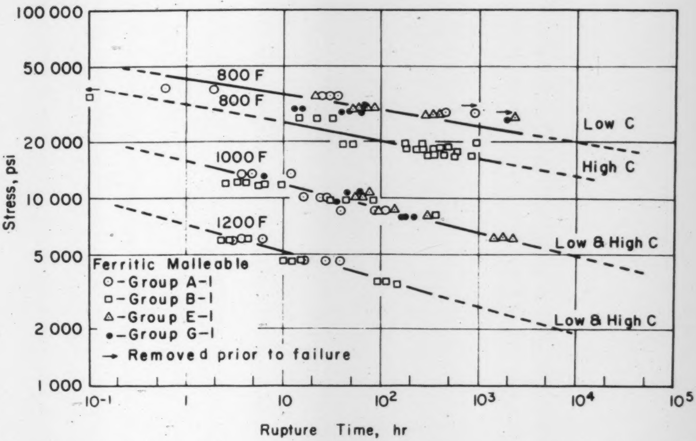

FIG. 3.—Stress-Rupture Plot for Ferritic Malleable Iron. At 800 F high carbon and 1000 F points which actually occupy the same level of stress have been displaced slightly in order to show all the data.

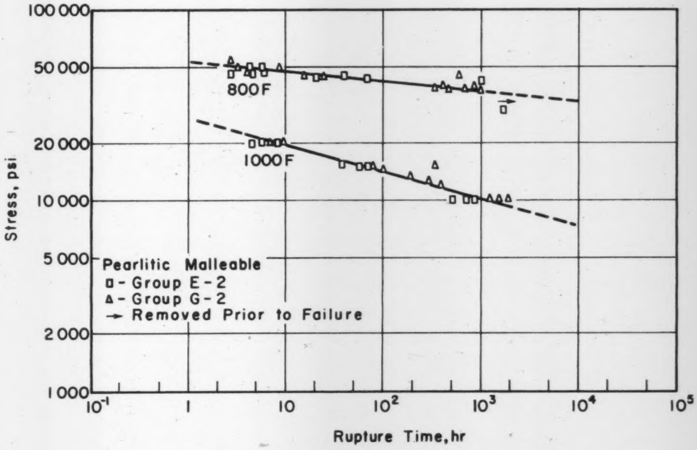

FIG. 4.—Stress-Rupture Plot for Pearlitic Malleable Iron.

A further verification is planned with precise extensometer equipment, measuring between gage marks.

TEST RESULTS

Figure 3 gives stress-rupture plots for low-carbon ferritic malleable irons at 800 F, high-carbon ferritic iron at 800 F, and both high- and low-carbon ferritic malleable irons combined at 1000 and 1200 F. The coordinates are log stress, S, *versus* log rupture time in hours. Data fit a straight line closely in this form of presentation. Lines drawn in the figure are calculated least squares fits to the data, employing a standard regression

TABLE II.—CALCULATED VALUES OF STRESSES FOR TIMES TO RUPTURE INDICATED FROM DATA OF FIGS. 3, 4, AND 5.

(Extrapolated values for 10,000 and 100,000 hr are provisionally included.)

Group	Number of Observations	Rupture Time, hr	Initial Stress, psi	Minimum Rupture Time, 95 per cent Confidence, hr[a]
Calculations from log-log data:				
Ferritic (low-carbon) groups A-1, E-1, G-1 at 800 F (Fig. 3)	21	1	42 150	. . .
		1 000	24 400	135
		10 000	20 350	990
		100 000	16 950	15 050
Ferritic (high-carbon) group B-1 at 800 F (Fig. 3)	21	1	31 000	. . .
		1 000	17 000	280
		10 000	14 000	2 580
		100 000	11 400	21 900
Ferritic (high- and low-carbon) groups A-1, E-1, G-1, B-1 at 1000 F (Fig. 3)	35	1	15 700	. . .
		1 000	6 650	480
		10 000	4 990	4 470
		100 000	3 750	41 000
Ferritic (high- and low-carbon) groups A-1 and B-1 at 1200 F (Fig. 3)	15	1	7 350	. . .
		1 000	2 680	335
		10 000	1 920	2 550
		100 000	1 370	18 600
Pearlitic groups E-2 and G-2 at 800 F (Fig. 4)	23	1	52 460	. . .
		1 000	37 140	130
		10 000	33 100	1 050
		100 000	29 500	7 750
Pearlitic (low-carbon) groups E-2 and G-2 at 1000 F (Fig. 4)	20	1	27 400	. . .
		1 000	10 300	380
		10 000	7 380	3 450
		100 000	5 320	29 900
Calculations from Larson-Miller data:				
Ferritic (high-carbon) group B-1 at 800 F (Fig. 5)	71	1	33 500	. . .
		1 000	16 600	. . .
		10 000	13 200	. . .
		100 000	10 400	. . .
Ferritic (high- and low-carbon) groups A-1, E-1, G-1, B-1 at 1000 F (Fig. 5)	71	1	15 500	. . .
		1 000	6 890	. . .
		10 000	5 260	. . .
		100 000	4 010	. . .
Ferritic (high- and low-carbon) groups A-1 and B-1 at 1200 F (Fig. 5)	71	1	7 180	. . .
		1 000	2 850	. . .
		10 000	2 100	. . .
		100 000	1 540	. . .

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[a] Calculated from actual data by standard regression analysis and based on sample number shown in column 2.

analysis. The calculations also yield values of the intercepts at 1, 1000, 10,000, and 100,000 hr. On this basis extrapolations have been shown by dotted line in Figs. 3 and 4 to 10,000 hr. The calculated 100,000-hr values are given in Table II and discussed in a later section of this paper.

The data obtained thus far with the standard malleable irons indicate a general high level of stress *versus* rupture time, equal or superior to ferritic cast materials for which data are available, particularly at 800 F. There is no evidence in any of the experimental results of abrupt changes in behavior or structure during the test periods, which extend from approximately 1 to over 2000 hr. All the data thus far exhibit a good linear fit on the log-log plots, with no breaks in slope of the curves as might be expected if fundamental changes are taking place. Metallographic examination confirms this with respect to microstructure. This renders the material useful in special applications such as ovens, driers, some types of furnaces, and many kinds of high-temperature fittings, both structural and plumbing.

In Fig. 3, data for both low- and high-carbon ferritic malleable irons appear to group closely at 1000 and at 1200 F as well. The methods of analysis employed give no certain indication of a significant difference in the behavior of the materials. More data are desirable at the longer times, particularly at 1200 F. With this caution in mind there appears ample justification, for the time being, for regarding both materials as identical in high-temperature stress-rupture properties. The least squares fits have been calculated from the combined data for these temperatures.

At 800 F, however, a distinct separation favoring the low-carbon malleable iron appears between the data for the two materials. The high-carbon malleable iron exhibits considerably shorter life at comparable stresses for all test periods covered. Allowable working stresses for this material must, therefore, be reduced in comparison to the low-carbon malleable iron, as shown in Table II. Still, the high-carbon malleable iron has considerable promise for use in load-carrying applications at 800 F.

The low-carbon malleable iron at 800 F appears, however, to be outstanding in characteristics. The least squares log-log curve fitting the data has a much lower slope than might be expected for this temperature from comparison with the data at 1000 and 1200 F. A few points obtained at 860 F, not shown in this paper, also give some indication that at the latter temperature, behavior of the high- and low-carbon material may still be identical. In any event, tests of specimens from three producers, which all agree as indicated by the curve in Fig. 3, yield strong evidence that low-carbon malleable iron at 800 F, and for an undetermined range below, may be an entirely different material in mechanical and physical characteristics. The methods of these experiments yield no direct evidence concerning the nature of these differences, highlighting the stress-rupture properties at elevated temperatures. Further work is needed and will be carried out. It is possible that electron micrographs employing new carbon replica techniques, with the attendant improvement in resolution limit to 20 Å or better, may yield information of value concerning possible changes in the microstructure over these ranges of experimental parameters.

Figure 4 presents the data obtained thus far with two groups of low-carbon pearlitic malleable irons. Analyses are given in Table I. Both are from different suppliers, produced by the method of arrested first-stage graphitization following a small manganese ladle addition. These meet the tensile requirements of the first five grades of pearlitic malleable iron of ASTM Specification A 220.[5] One, G-2, falls very close to the requirements of grade 60003 and is borderline only in yield strength. At 1000 F, rupture stress levels appear superior to the ferritic malleable irons at the same temperature, but lie slightly under high-carbon ferritic irons at 800 F.

Points obtained thus far with pearlitic irons at 800 F are plotted in Fig. 4. A least squares curve through these points indicates minimum rupture stress superior and parallel in trend to the low-carbon ferritic material at the same temperature (see Fig. 3).

The values obtained for these pearlitic malleable irons compare very favorably with those published for molybdenum alloyed irons (**5**) by Scholz, Doane, and Timmons. Here stress values can be compared tentatively at 1100 F by use of Larson-Miller extrapolations. On this basis, the pearlitic irons in this study appear equal to alloyed high-carbon materials containing as much as 0.5 per cent molybdenum, which had been added to enhance the characteristics by stabilizing the carbide phase. In other comparisons, the pearlitic malleable irons tested appear to be superior to other unalloyed cast pearlitic irons and equal or superior to most alloyed cast irons (**7**).

Methods of Interpretation:

In addition to the results mentioned above, the usefulness of these data has been extended through application of statistical methods to two types of interpretation: (1) linear log-log plots and (2) Larson-Miller type analysis. Both methods involve assumptions which are common in part to both. These fall into two categories: (1) those related to statistical considerations, and (2) those pertaining to physical and structural phenomena.

Log-Log Plots.—Table II compares the properties of all the pearlitic and ferritic materials tested thus far on the basis of the log-log plots (Figs. 3 and 4). Stress levels given are the calculated intercepts at 1, 1000, 10,000, and 100,000 hr by least squares methods from the data. Values for low-carbon and high-carbon ferritic malleable irons at 800 F are given separately. Those for low- and high-carbon ferritic irons at 1000 and 1200 F were determined from the combined data as in the case of the curves of Fig. 3.

Minimum rupture time values, at a 95 per cent confidence level, are also shown for some of the data for log-log plots in Table II. These apply to Figs. 3 and 4. The limits result from calculations based on standard regression analysis of the data. In general, the minimum values are lower for smaller samples because the statistics automatically take into account the meager knowledge and are forced to predict a lower value to maintain the 95 per cent probability of being right.

In applying the above, several assumptions related to statistical considerations must be kept clearly in mind:

1. The calculated parameters are estimates of the true parameters, not the true parameters themselves.
2. The results hold only for that population of specimens from which the test specimens are a true random sample.
3. The value of log t calculated from the equation for a given stress is an average value. About half the measured values will be above this, about half below.
4. The deviations of measured from calculated average values are assumed to be random and not a function of stress or time. Therefore scatter of points about the line is uniform. For example, the scatter at the high end of the line is no more and no less than at the low end.
5. Calculations of confidence levels have been made using the Student's t

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[5] 1958 Book of ASTM Standards, Part 1.

distribution, which assumes that the above deviations for the entire population would fit a Gaussian or normal distribution, on a logarithmic time scale.

In addition, the assumption is made that structurally there are no new or discontinuous phenomena occurring at times between the longest test time and the value for which extrapolation was made.

Larson-Miller Plots.—A study has been made of Larson-Miller plots, sometimes also called master rupture curves (8). These relate the three parameters: stress, temperature, and time on the basic assumption that the materials follow a time-temperature law of the form:

$$\dot{\epsilon} = Ae^{-Q/RT} \qquad \text{(1)}$$

where:

$\dot{\epsilon}$ = process rate,

A = a constant,

Q = process activation energy,

R = gas constant, and

T = absolute temperature.

If the assumption is made that the process rate is a constant and A is properly defined, then it can be shown that:

$$T(C + \log t_c) = \text{constant} \quad \text{(independent of stress)} \qquad \text{(2)}$$

where:

$C = \log A$ = another constant, and

t_c = time for completion of the process.

Larson and Miller assert that C is also independent of stress and show curves of:

$$\log \text{stress } \textit{versus } T(20 + \log t) \times 10^{-3} \qquad \text{(2a)}$$

where:

T = Rankine absolute temperature,

t = time to rupture in hours, not the continuous variable time in time elongation studies, and

C = 20.

C was found to be about 20 for several metals. In much of the literature C has been determined graphically. Stress-rupture data are often presented in the form of such Larson-Miller plots and extrapolations quite often made from them. Such extrapolations rest on the assumption that the line which fits the data in the known region can be extended and that the extension will continue to fit the data where none are readily available (for example, for different times or temperatures) and where checking the extrapolation is virtually impossible. In order for this assumption to be true it is very likely that:

1. There are no sudden physical changes in the material during the time to rupture. This assumption is also made in log-log extrapolation.
2. No physical changes occur in the material between one temperature level and another, represented in the data or used in the extrapolation. This assumption is unique with the Larson-Miller interpretation.

The Larson-Miller analysis is chiefly of value in estimating rupture stress of the material at temperatures other than those represented by the data. Time and temperature extrapolations may be of doubtful value unless the above criteria are taken into account. The range of parameters covered in this paper for some materials is large enough to reduce materially the uncertainties in extrapolation, both as regards the form of the curve and the accuracy with which the curve itself is known. In general, however, caution should be exercised in applying Larson-Miller plots beyond the time and temperature range of the data.

The authors have applied a "least squares" method to compute the parameters for a Larson-Miller plot. The assumptions and main steps involved in the analysis are shown below.

If the data satisfy the requirement that a Larson-Miller plot is a straight line (except for random scatter), then log stress and $T(C + \log t) \times 10^{-3}$ are related by a linear equation:

$$\log S = \frac{1}{\mu}\,[T(C + \log t) \times 10^{-3}] + \frac{\lambda}{\mu} \quad ..(3)$$

where:

$\frac{1}{\mu}$ = slope of the line,

$\frac{\lambda}{\mu}$ = log S intercept,

$T(C + \log t) \times 10^{-3}$ = abscissa (see Eq 2*a*),

S = initial stress to cause rupture,[6] and

= time to rupture (see Eq 2*a*).

When the data are taken, temperature and stress are independent variables and time is the dependent variable.

Solving Eq 3:

$$\log t = \lambda\left(\frac{1000}{T}\right) + \mu\left(\frac{1000}{T}\log S\right) - C \quad ..(4)$$

Let t_k, T_k, and S_k be the values for the variables for the k^{th} data point $(1 \leq k \leq N)$. Then a least squares solution requires that: $\sum_{1}^{N} {}_k(\log t - \log t_k)^2$ be a minimum by varying λ, μ, C. Standard calculus methods result in three simultaneous linear equations involving sums of the functions of T_k, S_k, and t_k. These are solved by methods suitable for calculating machines (9). Confidence limits for rupture time at any stress and temperature can then be established (10). The uncertainties (standard deviation) for λ, μ, and C can also be calculated from the same data (10).

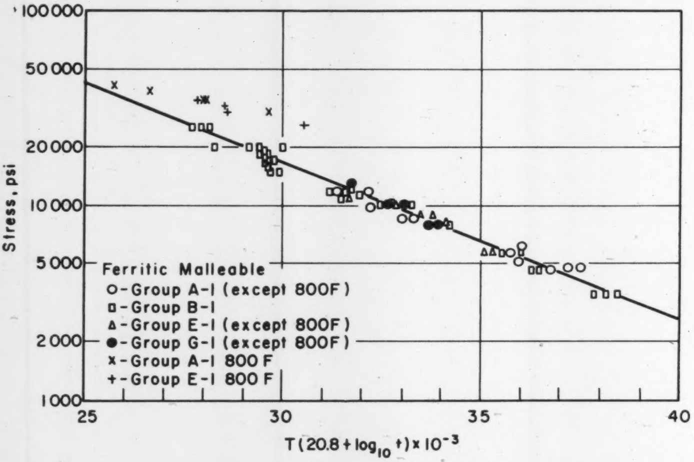

FIG. 5.—Larson-Miller Type Plot for Ferritic Malleable Iron. Note use of 20.8 value for the constant. Low-carbon malleable iron data at 800 F (groups A-1, G-1) are shown for comparison, but were not used in calculating the 20.8 value. Note also staggering of some points as in Fig. 3 to accommodate all the data. T = Temperature, deg Rankine ; t = rupture time, hr.

The same five statistical conditions mentioned previously with the log-log analysis apply to the above Larson-Miller interpretation. In addition, the deviation, in assumption 4, is considered independent of temperature. In general these assumptions seem to be reasonably true, at least insofar as the results have been used. It should be noted that the uncertainty in both interpolated and extrapolated values can be calculated and is frequently larger than expected. It is made of several parts: the uncertainty in estimating the parameters (λ, μ, and C for Larson-Miller plots) and the natural random spread in rupture time values under any one set of conditions.

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[6] If there is reduction of area during the creep process, the stress will increase since the force is constant. This is the initial value of the stress.

Employing this method, a value of $C = 20.8$ has been obtained from the combined data for high- and low-carbon ferritic malleable iron at 800, 1000, and 1200 F. Values for low-carbon ferritic irons at 800 F have been omitted from this calculation because they are believed to be representative of a different material and thus do not satisfy the original criteria above for this analysis. Figure 5 shows a plot of log stress *versus* $[T\ (20.8 + \log t) \times 10^{-3}]$. Calculated intercepts from these data for 1, 1000, 10,000, and 100,000 hr at 800, 1000, and 1200 F agree with those obtained from the log stress *versus* log rupture time diagrams. These are included in Table II for comparison.

Trial computations similar to those of Fig. 5 have been made for some of the other materials in Table II. Values of C range from 17 to 23.5. A Larson-Miller constant of 23.5 derived for group B-1 (high-carbon ferritic) also furnishes good agreement with the log-log presentation of Fig. 3. The use of this constant should provide reasonably reliable results because of the relatively large sample number of 40 for this group. Where data groups exist for only two temperatures, over an insufficient range of time, or the data indicate association with distinctly altered characteristics of the material as for the low-carbon ferritic malleable iron at 800 F, the agreement with the log-log plots is generally poor. It is anticipated that further studies of this nature will be published later in cases where the analytical methods can be applied.

Because of the statistical uncertainties entering into the calculation, the Larson-Miller form of presentation has been made only for the one case in Table II and Fig. 5. Confidence limits derived from these calculations apply to uncertainties in the constants λ, μ, and C, Eqs 3 and 4 above. For example, the 90 per cent confidence limits for the value of $C = 20.8$ in Fig. 5 are 19.5 to 22.2. The Larson-Miller extrapolations in Table II (particularly to 100,000 hr)

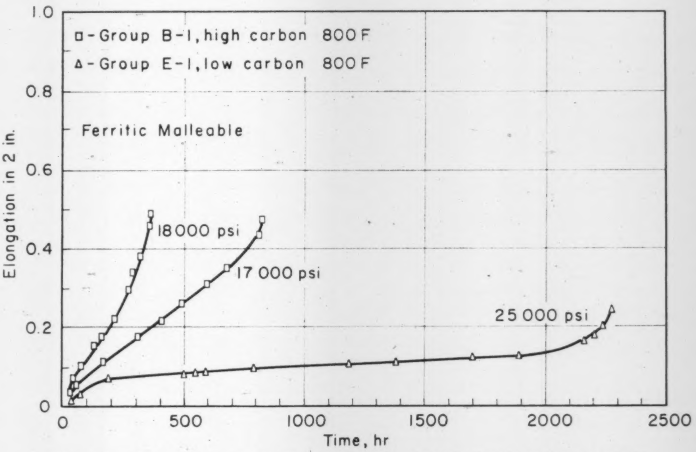

FIG. 6.—Time-Elongation Curves for Ferritic Malleable Iron at 800 F.

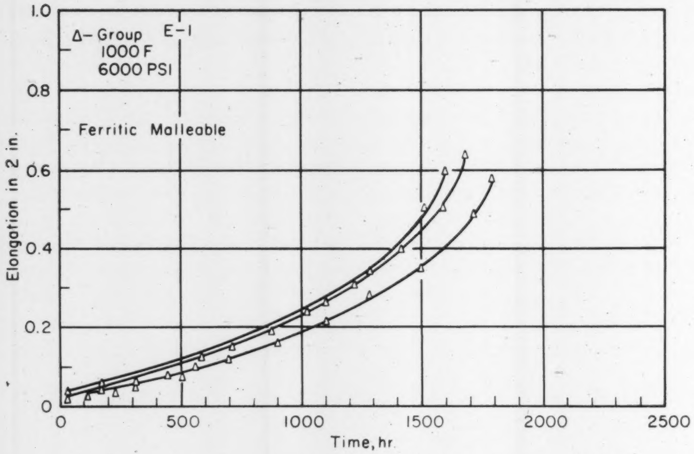

FIG. 7.—Time-Elongation Curves for Low-Carbon Ferritic Malleable Iron at 1000 F and 6000 psi.

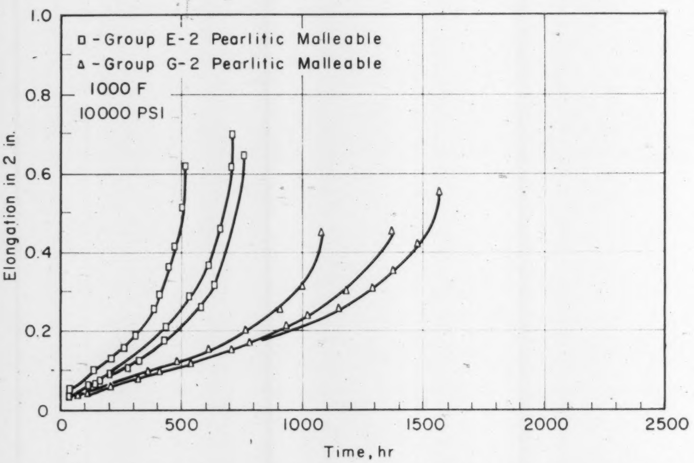

FIG. 8.—Time-Elongation Curves for Two Groups of Pearlitic Malleable Iron at 1000 F and 10,000 psi.

are subject to considerable uncertainty and are primarily for comparison with results published for other materials.

The analytical methods employed here for reduction of log-log and Larson-Miller data and plotting of curves have one distinct advantage over purely graphical methods. They can be applied with the conviction that false conclusions due to lack of knowledge of the uncertainties involved are unlikely. Statistical methods apply to small as well as to large groups of data. Generally, in the case of few data, the calculated uncertainties in the results turn out to be rather large. These uncertainty calculations and evaluation of confidence limits result from the computations with little additional effort.

Naturally the uncertainties in extrapolated values are reduced as more data are obtained. This stresses the need for data in adequate quantities, which is also borne out by the published work of Phillips and Sinnott (11).

TIME-ELONGATION MEASUREMENTS

Typical time-elongation curves for several groups of ferritic and pearlitic malleable irons appear in Figs. 6, 7, and 8. These clearly exhibit the three stages frequently reported in the literature (12,13). In some of the curves the first stage is not fully drawn because of the time scale that would be required. The normally ductile behavior of both pearlitic and ferritic malleable iron at all times and temperatures investigated is emphasized in these figures. No significant change in either elongation or reduction of area with time has been observed. Variations with temperature occur as described (Figs. 1 and 2) for elevated temperature short-time tension tests reported earlier in the paper. The curves in Fig. 6 illustrate changes which occur in the shape of the time-elongation curve at different loads for two groups of ferritic specimens. Values obtained for the minimum creep rate (linear or second stage portion) vary from 0.0017 per cent per hr for long rupture times to 4 per cent per hr at the highest stress levels and short times for the various conditions included in this study. It should be noted in Fig. 6 that the long-time test at 25,000 psi is from a different group than the other two curves shown. These two groups showed different elongation values at 800 F so that it is not to be concluded that any loss of ductility occurred in the longer time tests.

Figures 7 and 8 illustrate time-elongation behavior at 1000 F for both ferritic and pearlitic material. The same general type of curve is obtained at all temperatures investigated and some generalized approximations appear warranted on the basis of these tests. Approximately 50 per cent of the elongation occurs in the first two stages of creep, which frequently account for the useful life of a part in service. The third stage is generally so rapid that a part would not be useful under such conditions. This 50 per cent elongation generally occurs in approximately 70 per cent of the time to failure. The latter time value ranges in these experiments from approximately 60 per cent at higher loads and temperatures to somewhat over 80 per cent for longer time tests.

Extension measurements actually represent the sum of elongation due to creep as well as that which might result from growth of the specimen such as might be caused by oxidation. Examination of microstructures and measurements on test specimens indicate that growth is not significant.

The rapid elongation occurring in the third stage of creep is assumed to be caused by the increase in stress resulting from reduction of area because of the extremely ductile behavior exhibited by malleable iron. It has been pointed out by Parker (13) that the third stage portion can be obtained as the result of structural changes from materials exhibiting rather low elongation. This does not appear to be the case with either pearlitic or ferritic malleable iron.

METALLOGRAPHY

Extensive metallographic examination has been carried out on all specimens. Failed test specimens are sectioned both longitudinally and transversely in the area of fracture. Although it is difficult to be certain that differences observed

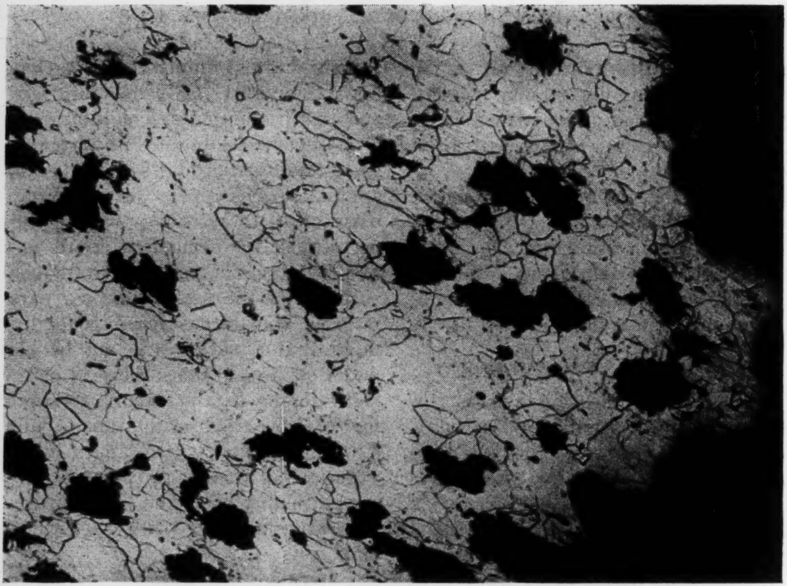

Fig. 9.—Transcrystalline Fracture of Low-Carbon Ferritic Malleable Iron (Group A-1). Specimen failed after 475.4 hr at 800 F and initial stress of 35,000 psi. One per cent nital etchant (×100).

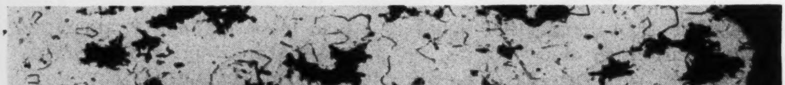

Fig. 10.—Transcrystalline Fracture of High-Carbon Ferritic Malleable Iron (Group B-1). This specimen failed after 868.0 hr at 800 F and initial stress of 17,000 psi. One per cent nital etchant (×100).

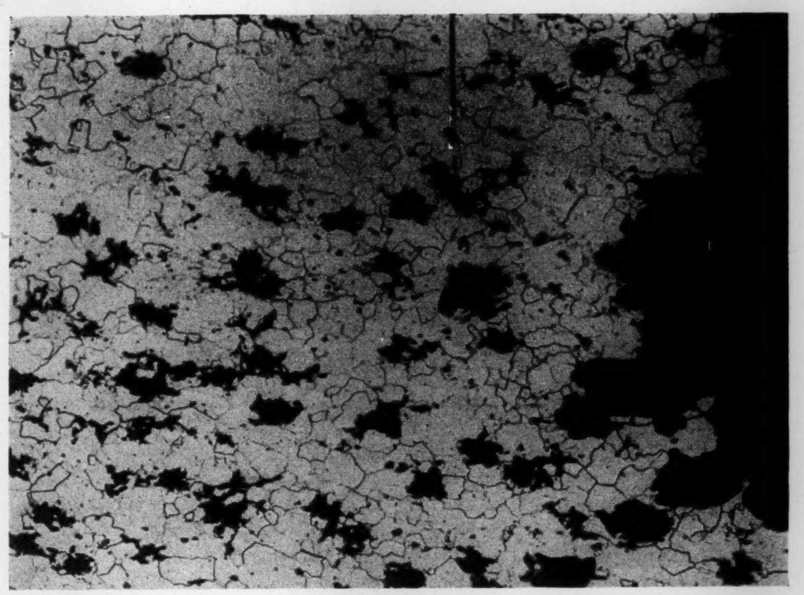

FIG. 11.—Intercrystalline Fracture of High-Carbon Ferritic Malleable Iron (Group B-1). Specimen failed after 128.3 hr at 1200 F and initial stress of 3600 psi. Etched in 1 per cent nital (×100).

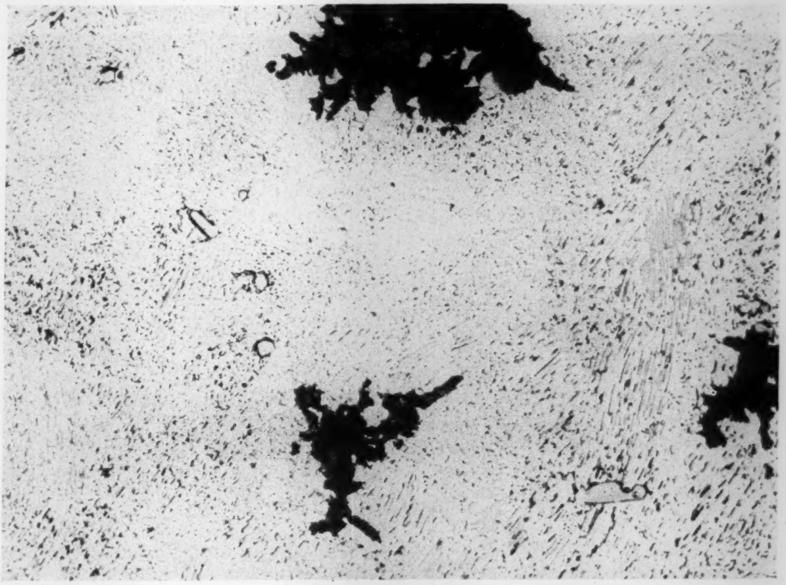

FIG. 12.—Typical Original Structure of Pearlitic Malleable Iron (Group G-2). Etched in 1 per cent nital (×500).

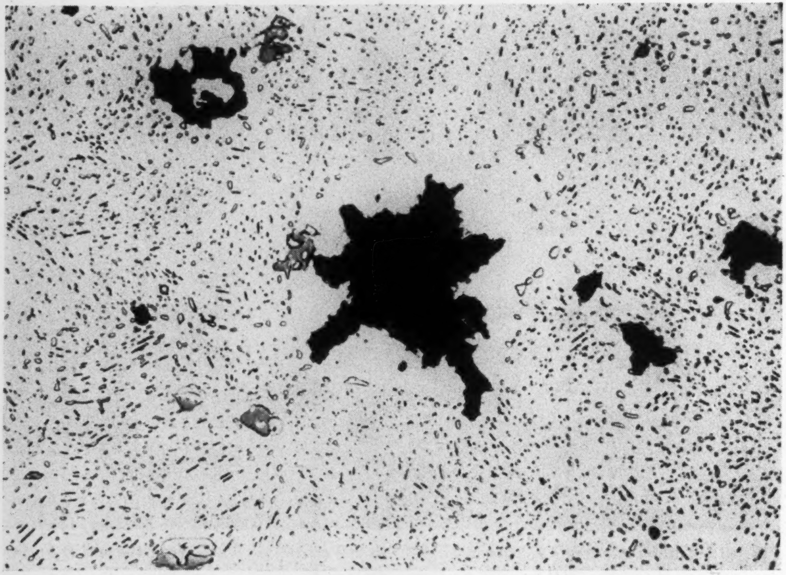

FIG. 13.—Typical Original Structure of Pearlitic Malleable Iron (Group E-2). Etched in 1 per cent nital (×500).

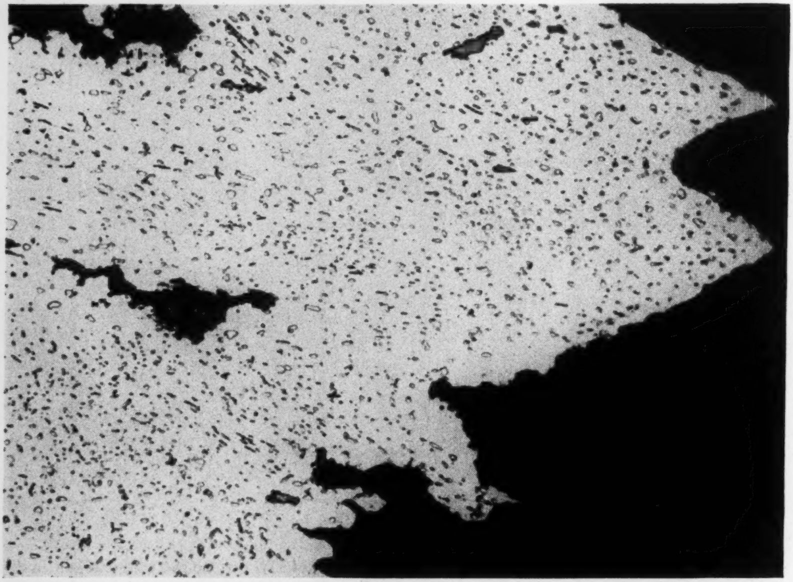

FIG. 14.—Failed Area of a Pearlitic Malleable Specimen (Group E-2) After Exposure to 1000 F for 754 hr and an Initial Stress of 10,000 psi. Etched in 1 per cent nital (×500).

in photomicrographs are truly representative, every effort has been made to show areas that are typical of results observed repeatedly on many specimens. For the sake of brevity only six photomicrographs are included in this paper, but it is believed they adequately represent some of the more important features observed in the study. A comparison of Figs. 9 and 10 illustrates the difference between one low-carbon ferritic malleable iron (group A-1), and high-carbon iron (group B-1). The higher carbon material shows more temper carbon nodules, which appear to be somewhat more finely dispersed than those in the low-carbon specimen. The high-carbon nodules also appear more irregular (or "sprawly"). Both materials at 800 F show considerable distortion in the vicinity of the fracture. The fracture surface is shown at one side of each photomicrograph.

Figure 11 shows a high-carbon specimen (group B-1) after failure at 1200 F. In this case, failure occurs typically in the ferrite grain boundaries. Little or no distortion of the ferrite grains and temper carbon nodules is observed compared to failures at 800 F (Figs. 9 and 10). The fractured surface in Fig. 11 also shows what is apparently a layer of iron oxide resulting from exposure at the high temperature after failure. The observation that failure shifts from transcrystalline to intercrystalline with increasing temperature agrees with early observations by Rosenhain and Ewen (**14**), Beilby (**15**), and Jeffries (**16**). Jeffries employed the term "equi-cohesive" temperature to describe that temperature at which the grain strength and grain boundary strength are equal, and failure can occur in either or both areas. Extensive observations indicate that change of failure from grains to grain boundaries occurs over a range of temperatures approximately from 800 to 1000 F for the malleable irons studied. Although differences in structure are apparent between the high- and low-carbon ferritic malleable irons, no explanation is apparent at the moment for the marked difference in performance at 800 F.

Figures 12 and 13 show typical structures prior to exposure at high temperature for two pearlitic malleable irons (groups E-2 and G-2) employed in this investigation. It should be emphasized that some differences exist between and within specimens of a given group so that photomicrographs shown can only be considered as typical. Both materials contain temper carbon nodules, carbides with varying degrees of spheroidization and gray manganese sulfide inclusions. Data from both groups are included in Fig. 4.

It is difficult to determine the extent of graphitization, if any, occurring during prolonged exposure to elevated temperatures. Depending upon what particular specimen and areas are selected, quite different conclusions may be drawn. It is believed, however, that the typical area of an E-2 specimen exposed to 1000 F at 10,000 psi for 754.4 hr indicates the extent of graphitization is quite small. Comparison of Fig. 14 with Fig. 13 shows very similar amounts of the carbide phase. Depletion of carbide around the graphite in Fig. 13, of course, results from the method of manufacture rather than test conditions, since this is the structure of a specimen prior to testing.

CONCLUSIONS

1. These measurements with ferritic and pearlitic malleable irons of standard composition yielded data which make it possible to estimate levels of stress applicable at high temperatures.

2. Data on the ferritic materials at 800 and 1000 F are regarded as sufficient in quantity for determination by statistical methods of loads carried in engineering applications.

3. There appears to be a distinct separation in performance at 800 F exhibited

by low-carbon ferritic malleable iron, which is considerably superior in load capacity at this temperature to high-carbon material.

4. Metallographic examination at standard magnifications with conventional methods yields no information bearing upon the above difference in behavior. It is possible that contemplated studies with the electron microscope will produce important clues.

5. The high- and low-carbon ferritic malleable irons studied are indistinguishable in performance at 1000 and 1200 F.

6. The data on ferritic malleable iron at 1200 F indicate that satisfactory load capacity exists for extended periods of time. Steps are being taken to extend the range of data at this temperature so that specification load values can be achieved.

7. The data on pearlitic malleable irons indicate, for some grades, a high load capacity at 800 and 1000 F, somewhat greater than for the ferritic malleable irons.

8. Metallographic examination reveals no significant changes in structure or character of the pearlitic malleable irons thus far tested for ranges of time over 2300 hr. Graphitization may occur with some grades, depending upon the analysis of the iron and the processing methods employed. This is currently being investigated further.

9. Data for all the irons tested thus far, both ferritic and pearlitic, yield no evidence of fundamental changes in structure or physical characteristics. This is substantiated both through metallurgical examination and the fact that various plots of the data including log-log and Larson-Miller presentations show no evidence of breaks in the curves at the longest times.

10. It is believed that extrapolations based upon least squares calculations for the log-log plots of the data constitute a reliable index of the average performance to be expected from commercial malleable irons. Larson-Miller plots are particularly useful for predicting or interpolating to obtain the performance of the material at other than test temperatures. Extreme caution should be applied in using Larson-Miller plots for extrapolating beyond the times and temperature covered by the data.

It is evident that considerable additional work is needed in order to complete the picture of the performance of malleable irons at elevated temperatures. This is currently proceeding with a study of pearlitic irons and will be extended as soon as practicable to include alloy malleable irons. It should be emphasized that all of the data presented in this paper are concerned with ferritic and pearlitic malleable irons representative of current foundry methods and production.

Acknowledgments:

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DISCUSSION

MR. J. J. KANTER.[1]—After hearing the presentation of this information which has been made available on the high-temperature properties of these irons, one is disposed to ask why malleable iron has not enjoyed a wider use at elevated temperatures. Perhaps we will have to look at some of the other properties and considerations which encouraged the engineers to make the rules and codes which govern constructions using malleable iron. In the ASME Boiler Code,[2] for instance, until a very few years ago none of the cast irons—gray iron or malleable—were permitted for use at any higher temperature than 450 F. Then came a movement to extend the temperature usage and on the basis of heat treatment the range was extended to 650 F for gray irons of balanced composition. The safety factor of 10 to 1 on a basis of the minimum specified tensile strength is still retained, however, for all the gray irons. However, in the case of malleable iron, the limitation still stands at 450 F provided 18 per cent elongation is met, and in effect the safety factor required is almost as great as that for gray iron. More recently the code committee has been asked to recognize the nodular cast irons, in which case the limiting temperature is still 650 F but on a basis of 5 to 1 safety factor on minimum specified tensile strength.

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[1] Director of Research, Engineering Division, Crane Co., Chicago, Ill.

[2] ASME Boiler Code, Am. Soc. Mechanical Engrs.

It would certainly appear from the information now before us that at least as good a basis of stress rating as has been accorded to the nodular irons ought to be considered for malleable irons.

MR. L. C. MARSHALL (*author*).—I feel that the data given here for the ferritic and malleable irons will stand up under rather close scrutiny and can be used for engineering calculations and applications. The data on pearlitic irons are going ahead, also data are being gathered on alloy types of pearlitic malleable iron and will be presented in future publications. In general we feel these malleable irons reported compare very favorably with other cast ferrous materials that are currently available for design purposes.

MR. D. V. DOANE.[3]—It is encouraging to see the results of the Malleable Founder's Society experimental program on elevated temperature properties of malleable iron. The application of statistical methods to the analysis of stress-rupture results should hasten the establishment of industry design standards for the use of malleable iron at elevated temperatures.

The writer would like to comment specifically with regard to the comparison of the data in the paper with the data obtained by his co-workers, Reference 5 of the paper. The statement is made in the paper that "the pearlitic irons in this study appear equal to the alloyed high-carbon materials containing as much as 0.5 per cent molybdenum, which had been added to enhance the characteristics by stabilizing the carbide phase." This writer would like to call attention to the fact that the authors' irons were tested in the arrested anneal condition (pearlitic matrix) while the writer's irons were tested in the "stabilized" condition (essentially a ferritic matrix). The only valid comparison exists between the authors' "high-carbon ferritic iron," Group B-1, and the writer's "stabilized" base iron (unalloyed) and this comparison is very favorable, as the authors infer.

Examination of Fig. 14 of the authors' paper reveals considerable stability of the carbide phase of the low-carbon pearlitic malleable iron after exposure, under stress, for 746 hr at 1000 F. Stability studies by the writer of high-carbon pearlitic malleable iron indicated almost complete absence of the carbide phase after 1000 hr exposure at 1000 F under no stress. This may be additional evidence of the advantage of lower carbon contents in malleable iron, to which the authors allude.

The writer was interested in conclusion 8 of the paper. A complete study of the structural stability of pearlitic malleable iron is needed, and the authors indicate such a study is in progress. We will be interested in reviewing the results of this study.

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[3] Metallurgical Supervisor, Climax Molybdenum Company of Michigan, Detroit, Mich.

TEMPERATURE AND TIME STABILITY OF M257 AND SAP ALUMINUM - ALUMINUM OXIDE ALLOYS*

BY W. S. CREMENS,[1] E. A. BRYAN,[2] AND N. J. GRANT[3]

SYNOPSIS

The 6 to 8 per cent Al_2O_3 (M257) alloy and the 10 to 14 per cent Al_2O_3 (SAP) alloy in the system $Al\text{-}Al_2O_3$ were cold worked up to 66 and 29 per cent, respectively, after which they were annealed for various periods of time near the melting point. The effect of cold work, annealing time, and temperature on the resultant tensile, yield, and ductility values are reported, and the effect on the 600 F stress rupture properties were examined.

It has been reported that the aluminum - aluminum oxide alloys show a drop in hardness when heated close to the melting point. Since such a change in hardness might be due to recrystallization, a phenomenon not observed in the past for the higher oxide alloys (greater than about 6 per cent), a study was undertaken to observe the changes in properties that might take place when such alloys are heated near the melting point. Furthermore, additional knowledge regarding the structural stability of such alloys would aid in the design of analogous alloys in other systems and by other techniques.

MATERIALS

The Aluminum Company of America provided samples of its M257 sheet material, which contains 6 to 8 weight per cent oxide. Three conditions of working were represented: hot rolled, 29 per cent cold rolled, and 60 per cent cold rolled. The hot rolling had been done at 800 to 850 F with reheating for each pass.

The Société Anonyme pour l'Industrie de l'Aluminium, of Switzerland, supplied samples of a grade of SAP that contains 10 to 14 weight per cent oxide, in the hot rolled conditioned. Hot-rolled M257 and SAP strips were cold rolled to give additional reduction steps as indicated below:

M257		SAP	
Condition, per cent cold worked	Thickness, in.	Condition, per cent cold worked	Thickness, in.
0	0.123	0	0.197
15	0.105	15	0.167
29	0.125	20	0.158
49	0.063	29	0.140
60	0.125		
66	0.042		

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* Presented at the Sixty-first Annual Meeting of the Society, June 22–27, 1958.

[1] Formerly Research Assistant, Massachusetts Institute of Technology; now Technical Liaison Office, Headquarters U. S. Air Forces in Europe, Wiesbaden (Germany).

[2] Formerly Student, Massachusetts Institute of Technology; now Metallurgist, Jones & Laughlin Steel Co., Pittsburgh, Pa.

[3] Professor, Department of Metallurgy, Massachusetts Institute of Technology, Cambridge, Mass.

Edge cracks appeared in M257 with 66 per cent reduction and in SAP with 27 per cent reduction, but it was found that 66 per cent cold-worked M257 strips and 29 per cent cold-worked SAP strips would yield crack-free tension specimens Since most of the strips were seriously bent, the M257 strips were heated to about 300 F for 10 min and the SAP strips to 400 F for 15 min for hot straightening.

TABLE I.—HARDNESS (ROCKWELL F SCALE) OF M257 AS A FUNCTION OF ANNEALING TIME AND TEMPERATURE.

Per Cent Cold Work	As-rolled Hardness	At 1140 F		At 1180 F		At 1200 F		At 1210 F	
		Time, hr	Hardness	Time, hr	Hardness	Time, hr	Hardness	Time, hr	Hardness
66	77	2	74	2	75	3	70	...	...
	...	20	71.5	19	69	50	66	...	...
	...	51	69	65	69	...	...	...	...
	...	118	69	202	71	...	...	...	...
	...	235	70	...	...	...	...	...	...
60	78	...	...	...	...	...	...	2	65
	...	...	...	...	...	18	73	17	60
	...	...	...	...	...	49	75	26	53
	...	...	...	...	...	170	69	46	47
49	76	...	...	...	...	...	...	...	...
29	76	...	...	...	...	...	...	2	71
	...	...	...	...	...	...	...	17	66
	...	...	...	...	...	...	...	26	66
	...	...	...	...	...	...	...	47	65
	...	...	...	...	...	...	...	97	47
15	73	...	...	...	...	...	...	...	...
0	71	...	...	...	...	...	...	2	71
	...	...	...	...	...	...	...	17	65
	...	...	...	...	...	...	...	26	57
	...	...	...	...	...	...	...	47	61
	...	...	...	...	...	...	...	97	59

EXPERIMENTAL PROCEDURE

As a means of selecting the highest possible exposure temperatures for the studies of structure and property stability, preliminary hardness measurements were used. Specimens about 0.50 in. square were cut and then sealed under vacuum in Vycor tubes. Specimens were held at the temperatures for time periods shown in Tables I and II. The Rockwell F scale was used.

Tension test specimens were cut parallel to the rolling axis, except for a few which were cut across the rolling direction to study the transverse properties. At first the specimens were of the 0.50-in. wide by 2.25-in. long gage section, but because of a shortage of material and a blistering problem which arose, smaller specimens were substituted (0.160-in. wide by 1.25-in. long gage section). In spite of the blistering, the interpretation of the data was not too greatly hampered. While the change in dimensions of the test specimen caused a variation in elongation values, the yield

TABLE II.—HARDNESS (ROCKWELL F SCALE) OF SAP AS A FUNCTION OF ANNEALING TIME AND TEMPERATURE.

Per Cent Cold work	As-rolled Hardness	At 1140 F		At 1180 F	
		Time, hr	Hardness	Time, hr	Hardness
29	87	2	...	0.5	30
	...	20	86	21	0
	...	51	83	66	0
	...	124	84	...	...
20	86	...	...	0.5	30
	...	...	...	23	0
	...	...	...	40	...
15	84	...	...	...	...
0	84	...	...	...	...

TABLE III.—ROOM TEMPERATURE PROPERTIES OF M257 SHEET.

	Reported by Alcoa, As-Rolled			Annealing Time at 1200 F			
				22 hr	49 hr	100 hr	154 hr
Longitudinal							
Tensile strength, psi	**37 000**	**38 000**	36 000	**36 000**	...	**35 000**	...
Yield stress, 0.2 per cent	**29 000**	...	...	**25 000**	...	**22 000**	...
Elongation, per cent	**17**[a]	**14**	20	**20**	...	**16**	...
Transversal							
Tensile strength, psi	...	**39 000**	...	**31 000**	...	**37 000**	...
Yield stress, per cent	...	...	...	**21 000**	...	**24 000**	...
Elongation, per cent	...	**14**	...	**5**	...	**14**	...
29 Per Cent Cold Work							
Longitudinal							
Tensile strength, psi	**43 000**	**42 000**	43 000	**37 000**	37 000	**36 000**	34 200
Yield stress, per cent	**37 000**	...	...	**28 000**	...	**25 000**	...
Elongation, per cent	**8**	**6**	15	**12**	33	**14**	12.5
Transversal							
Tensile strength, psi	...	**44 000**	...	**35 000**	...	**37 000**	...
Yield stress, per cent	...	...	...	**24 000**	...	**26 000**	...
Elongation, per cent	...	**8**	...	**6**	...	**12**	...
49 Per Cent Cold Work							
Longitudinal							
Tensile strength, psi	...	...	44 000	...	...	...	36 700
Yield stress, per cent	...	...	...	...	...	...	...
Elongation, per cent	...	...	11	...	...	...	20
60 Per Cent Cold Work							
Longitudinal							
Tensile strength, psi	**44 000**	**44 000**	44 000	**37 000**	38 100	**35 000**	36 400
Yield stress, per cent	**36 000**	...	...	**30 000**	...	**26 000**	...
Elongation, per cent	**8**	**6**	10	**6**	23	**10**	22
Transversal							
Tensile strength, psi	...	**45 000**	43 000	**34 000**	38 000	**36 000**	...
Yield stress, per cent	...	...	...	**24 000**	...	**26 000**	...
Elongation, per cent	...	**8**	11	**4**[b]	19.5	**14**	...
66 Per Cent Cold Work							
Longitudinal							
Tensile strength, psi	...	...	42 000	...	37 700	...	34 400
Yield stress, per cent	...	...	37 000	...	...	...	...
Elongation, per cent	...	...	13	...	25	...	13

66 Per Cent Cold Work

				Annealing Time at 1180 F			
				63 hr	154 hr		
Longitudinal							
Tensile strength, psi	...	...	...	36 500	35 000	...	...
Yield stress, per cent	...	...	...	27 600	...	...	...
Elongation, per cent	...	...	...	19.0	19.0	...	...

[a] Data in boldface were obtained on 2 in. gage length specimens; other data was obtained on 1-in. gage length specimens.

[b] Severe blisters.

and tensile values were not affected. The tensile data are reported in Tables III and IV. All test data obtained on the larger specimens are underlined in Table III. The tension and stress-rupture specimens were not vacuum encapsulated for the temperature exposure tests. All tension tests were performed at room temperature.

Stress-rupture tests were made at constant load, at 600 F, on the two alloys in the following conditions:

- M257 (60 per cent cold worked), as cold worked,
- M257, (60 per cent cold worked), after 137 hr at 1200 F,
- SAP (20 per cent cold worked), as cold worked,
- SAP (20 per cent cold worked), after 23 hr at 1180 F.

The standard stress-rupture test unit was used with universal joints outside of the hot zone to assure uniaxial loading. Specimens used were 0.160 in. wide by 1.25 in. gage. Stress-rupture results are plotted in Fig. 4.

RESULTS AND DISCUSSION

The blisters were observed on annealing SAP above 1150 F and on M257 above 1200 F. The blisters ranged from pinpoint size to 15 to 20 mm in diameter in the several severe cases, and had blackened interior surfaces. Their occurrence is accompanied by a change from 45 deg shear, in sheet tension specimens, to a laminated, irregular fracture and by a drop and scatter in hardness values. Although most pronounced near the surface, they occur throughout the sheet thickness. The blisters were probably caused by gas adsorbed on or occluded by the original aluminum powder. Analyses for hydrogen showed the following:

	Hydrogen Content, ppm
SAP (0 per cent cold worked)	61
M257 (0 per cent cold worked)	45
SAP (20 per cent cold worked)	38
M257 (66 per cent cold worked)	38

These high hydrogen values are probably sufficient to explain the blisters. The lower hydrogen content of the cold-worked materials doubtless results from the heating they received prior to

TABLE IV.—ROOM TEMPERATURE TENSILE PROPERTIES OF SAP 29 PER CENT COLD WORKED. LONGITUDINAL SPECIMENS, 1-IN. GAGE LENGTH.

	As Cold Rolled	Annealing Time			
		At 1180 F[a]			At 1200 F[a]
		1.5 hr	23 hr	66 hr	170 hr
Tensile strength, psi	52 800	41 000	39 000	38 300	35 600
Yield strength, 0.2 per cent	38 400	...	33 400	32 700	...
Elongation, per cent	12.5	4.0	19.5	19.5	6.5

[a] SAP specimens were severely blistered at 1180 and 1200 F.

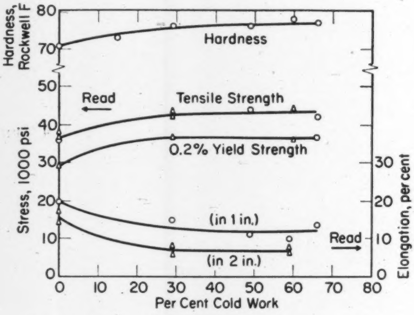

FIG. 1.—Change in Tensile Properties of M257 With Increasing Cold Work.

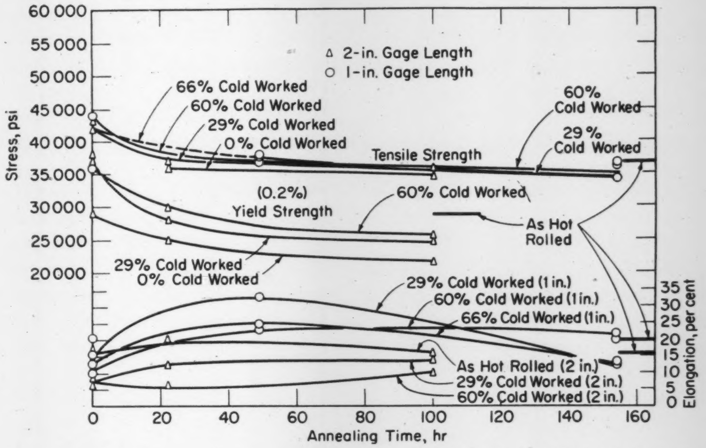

FIG. 2.—Change in Room Temperature Properties of Cold-Worked M257 with Annealing Time at 1200 F. Longitudinal specimens. Bars indicate as hot-rolled values.

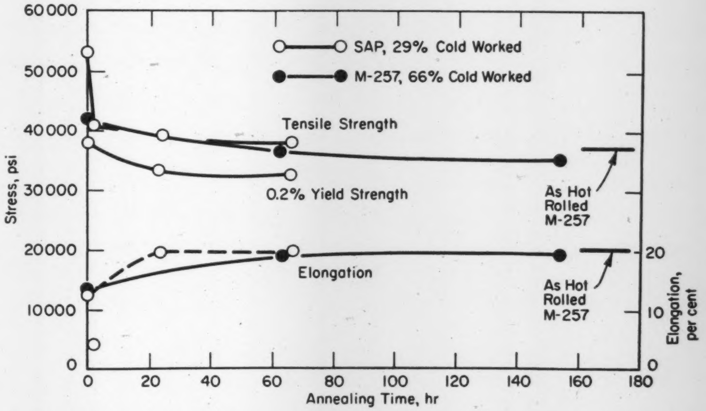

FIG. 3.—Change in Room Temperature Properties of Cold-Worked SAP and M257 with Annealing Time at 1180 F. Longitudinal specimens, 1-in. gage length.

straightening. It is possible that the blisters could be minimized or even eliminated by annealing in an inert gas, or in vacuum.

M257.—The effect of cold work on the tensile properties of M257 is shown in Fig. 1. Cold rolling to a 29 per cent reduction gives a 16 per cent increase in tensile strength and a 28 per cent increase in yield strength, while elongation falls to about half of its original value. Further reduction has no significant effect on properties.

In Fig. 1, only data for longitudinal specimens are plotted. Table III shows that there is little difference between longitudinal and transverse specimens in the as-rolled sheet prior to heating.

The hardness data of Table I show that the increase in hardness due to cold rolling can be annealed out at 1140 F, and possibly lower, but the hardness does not fall below the hot-rolled value until a temperature of 1200 F is exceeded and blisters appear.

Figure 2 shows the effect of static-annealing time at 1200 F on the room temperature tensile properties of M257. Within 50 hr, gains in tensile strength due to cold working have been annealed out, but after an additional 100 hr, all the tensile strength values are still within 7 per cent of the hot-rolled value. Yield strength values, however, fall significantly below the hot-rolled value. In this connection, it should be noted that some slight blistering was observed on the specimens held for 22 and 100 hr at 1200 F, and the yield strength values may be showing the effect of the blisters.

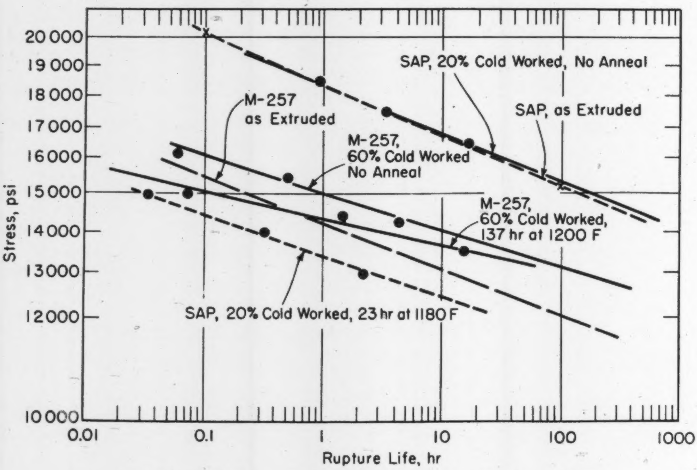

FIG. 4.—Stress-Rupture Results at 600 F for SAP and M257.

Elongation data in Fig. 2 show that there is a recovery with annealing time at 1200 F out to about 100 hr. In fact, the 1-in. gage length specimens show a maximum for the 49 hr treatment. Beyond about 120 hr, the 1-in. specimens show values that are less than for the as-hot-rolled specimens. The 2-in. specimens show slight recovery out to 100 hr, that of the 29 per cent cold-worked specimens approaching the as-hot-rolled value.

Conversely, it may be seen in Table III that transverse elongation values appear to go through a minimum value. This may be due to anisotropy introduced by incipient melting or blister formation. In Fig. 3, changes in tensile strength and elongation of 66 per cent cold-worked M257 with annealing time at 1180 F are shown. At this somewhat lower temperature, both the tensile properties and elongation reach the hot-rolled values in about 60 hr and level off.

SAP.—In Fig. 3 are plotted also the changes in properties of the 29 per cent cold-worked SAP as a function of annealing time at 1180 F. At that temperature severe blistering occurs in SAP and tensile strength is observed to drop sharply. Since elongation is sensitive to the presence of blisters in the gage length, the curve is drawn dotted. Additional points would be expected to give a wide scatter band. Table II shows that hardness drops sharply with as little as $\frac{1}{2}$ hr at 1180 F, while at 1140 F hardness falls only to the hot-rolled value. It was determined that blisters first become visible at 1155 F, and it is suspected that that temperature may also mark the solidus for the SAP alloy.

Stress-Rupture.—Data obtained at 600 F are plotted in Fig. 4. M257 (60 per cent cold worked) and SAP (20 per cent cold worked) were tested both as cold rolled and after the annealing treatments noted on the plot, and are compared to the curves for M257 and SAP in the as-extruded condition (bar stock). The 20 per cent cold work for SAP results in no change over as-extruded SAP tested at 600 F. The large decrease in strength due to the 1180 F anneal is due entirely to the blistering reaction. Alloy M257 shows a gain in strength with 60 per cent cold work over the as-extruded product. The remarkable stability of this alloy is noted by the maintenance of the high-temperature strength after an annealing treatment of 137 hr at 1200 F after the 60 per cent cold-work treatment. While the annealing treatment resulted primarily in a change of slope of the curve, this change is within experimental limits in view of the use of only 3 test points.

CONCLUSIONS

Only slight increases in hardness of M257 and SAP sheet were obtained with large amounts of cold rolling. These increases can be annealed out at 1140 F, and possibly at lower temperatures, but hardness falls only to the hot-rolled value for any temperature below that at which "blisters" appear.

"Blisters" were observed to form in SAP at 1155 F and in M257 at 1205 to 1215 F. They are attributed to gas evolution near the solidus temperature. An inert gas or vacuum annealing treatment is suggested as a method of eliminating the blistering.

It was shown for M257 that moderate increases in tensile and yield strength can be obtained with cold rolling, but as the hardness increases, they are annealed out at high temperatures without a significant loss of strength or ductility, unless blistering occurs.

The small effect of a 137 hr anneal at 1200 F upon the 600 F stress-rupture strength of M257, cold-worked 60 per cent, is a striking indication of the structural stability of the aluminum-aluminum oxide materials and of their resistance to structural change or property alteration.

Acknowledgments:

The authors are indebted to the Aeronautical Research Laboratory, Wright Patterson Air Force Base, for support of this program of research and the Aluminum Company of America and La Société Anonyme pour l'Industrie de l'Aluminium (AIAG), Switzerland, for supplying us with the alloys.

DISCUSSION

CHAIRMAN H. C. CROSS.[1]—Do I understand on the basis of the limited results to date that the authors would not recommend getting an improvement in rupture strength at 600 F by cold working of these particular materials? The curve differences are considered to be due to scatter, but on an extended scale there is really no difference between properties of the extruded and the cold worked M-257.

MR. OLIVER PRESTON[2] (*for the authors*).—Mechanical testing experience with this class of alloys would indicate that the stress rupture curves for the extruded and the cold-worked M-257 conditions are the same within experimental error limits.

CHAIRMAN CROSS.—This leads to the next question: The longest point on this curve for M-257 at 60 per cent cold work, no annealing, is a rupture life of about 6 hr. Six hours at 600 F is not a very long time. I wonder whether the authors have any later results at a longer rupture time than the maximum here of around 10 hr to see if there is any difference in slope of the cold-worked curve as compared with the extruded curve.

MR. PRESTON.—No, longer-time tests were not run, primarily because the long-time exposure tests of the cold-worked alloys just below the melting temperature did not produce a change in hardness or in tensile properties (compared to the extruded condition). For this reason, when the relatively short-time stress rupture tests showed similar slopes and test values at the much lower temperature of 600 F, it could be assumed with safety that these alloys would be stable even in long-time tests.

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[1] Assistant Technical Coordination Director, Battelle Memorial Institute, Columbus, Ohio.

[2] Research Assistant, Massachusetts Institute of Technology, Cambridge, Mass.

THE EFFECT OF ATMOSPHERE ON CREEP-RUPTURE PROPERTIES OF A NICKEL-CHROMIUM-ALUMINUM ALLOY*

By Paul Shahinian[1] and M. R. Achter[1]

Synopsis

The role of ductility in the effect of environment on creep and rupture properties was investigated employing a 76 per cent nickel, 19 per cent chromium, and 4 per cent aluminum alloy. Creep-rupture tests were conducted primarily in air and in vacuum at 1300, 1500, and 1900 F.

It was found that at low temperatures and high stresses time-to-rupture was longer in vacuum than in air, but at high temperatures and low stresses the reverse was true. Generally conventional minimum creep rates were unaffected by environment. An unusual creep curve was obtained in air at the higher temperatures, due apparently to oxidation, showing markedly greater than expected rupture time and ductility. The influence of atmosphere was related to the mode of crack formation in this relatively brittle material and can be explained by previously advanced mechanisms.

In previous investigations (**1, 2**)[2] it was shown that the influence of environment on creep and rupture properties of nickel and a nickel-chromium alloy depended upon temperature, strain rate, and oxidation resistance of the material. At high strain rates and low temperatures the metals were stronger in vacuum than in air while the reverse was true at low strain rates and high temperatures. That ductility may also be an important factor was indicated in studies on a notched material of low ductility (**3**).

This investigation was conducted with a relatively brittle nickel-chromium-aluminum alloy to determine the role of ductility and because it has possibilities as a cladding material for refractory metals. Furthermore, this ternary alloy serves as a base for many of the leading, more complex, high-temperature alloys, and therefore, the manner in which its properties are affected by test atmosphere may be used as a guide to predict the behavior of commercial alloys.

Experimental Procedure

The chemical composition of the aluminum-modified nickel-chromium alloy was as follows: 19.1 per cent chromium, 3.60 per cent aluminum, 1.7 per cent silicon, 0.54 per cent iron, 0.10 per cent manganese, 0.027 per cent carbon, 0.004 per cent sulfur, and the balance nickel. Sheet test specimens of 0.500-in. width and 1.25-in. gage length were machined from 0.033-in. thick strip material; the surface oxide was removed by polishing. Prior to testing, the specimens were given an anneal in vacuum at 2000 F for 2 hr which resulted in an average grain diameter of 0.036 mm. The treatment left the material in a brittle condition; large amounts of the Ni_3Al, γ' phase, which is described by Taylor and Floyd (3), were in the grain boundaries.

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* This paper was presented at the Sixty-first Annual Meeting of the Society, June 22–27, 1958.

[1] U. S. Naval Research Laboratory, Metallurgy Division, Washington, D. C.

[2] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 772.

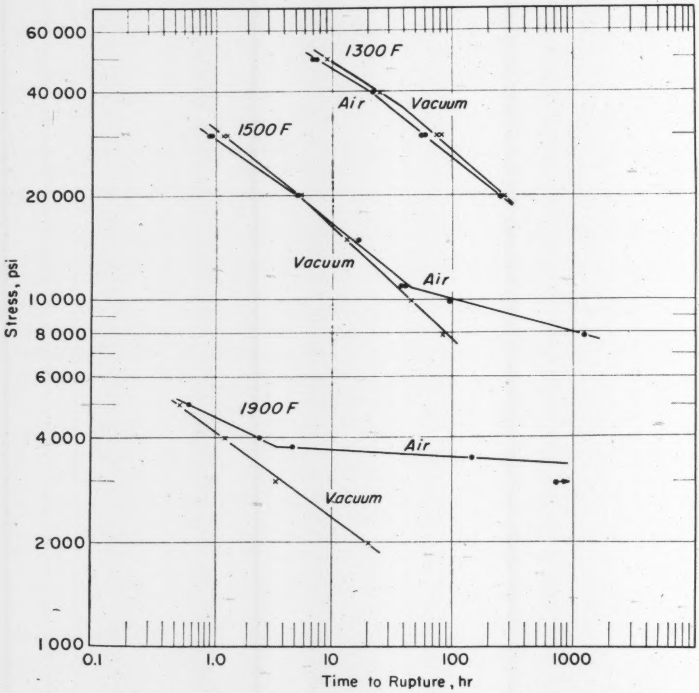

FIG. 1.—Variation of Rupture Time with Stress in Air and in Vacuum at 1300, 1500, and 1900 F.

Creep-rupture tests were conducted at several stress levels at 1300, 1500, and 1900 F primarily in air and in vacuum ranging from 1×10^{-5} mm of mercury at the lowest temperature to 2×10^{-4} at the highest. Several tests were also run in stagnant atmospheres of oxygen and of nitrogen. In addition, interrupted tests were run in order to examine microstructural changes during creep. The specimens were loaded in approximately 1 hr after reaching the desired temperature which was maintained to within ±2 F during the test. Extension of the specimen was measured with a dial gage attached to the upper pull rod outside of the atmosphere chamber. Elongation-at-fracture values were determined from gage marks scribed on the specimen. Additional details regarding the test equipment and procedure may be found in a previous publication (4).

Coupons of the alloy quenched from the test temperature revealed that, although the microstructure was unchanged at the lower temperatures, at 1900 F a large portion of γ' phase had gone into solution. Metallographic examination was also made of the fractured and interrupted test specimens. No evidence of grain growth during the tests were observed. Determinations of oxygen and nitrogen contents by the vacuum fusion method were made on the original material and some of the fractured specimens.

Test Results

Creep-Rupture Data:

The time-to-rupture data for both air and vacuum tests are plotted *versus* stress (on a log-log basis) in Fig. 1. It can be seen that rupture time may be longer in either air or vacuum depending on the test temperature and stress level. At 1300 F, rupture times were longer in vacuum while at 1900 F they were longer in air. Although a reversal point was observed only at 1500 F, the possibility of reversal points at the other two temperatures is indicated by extrapolation of the curves. The data of Fig. 1 may be divided into two areas; these will be referred to as the air-strengthened and air-weakened regions. In general, the data points for high temperatures and low stresses are located in the air-strengthened region while at low tem-

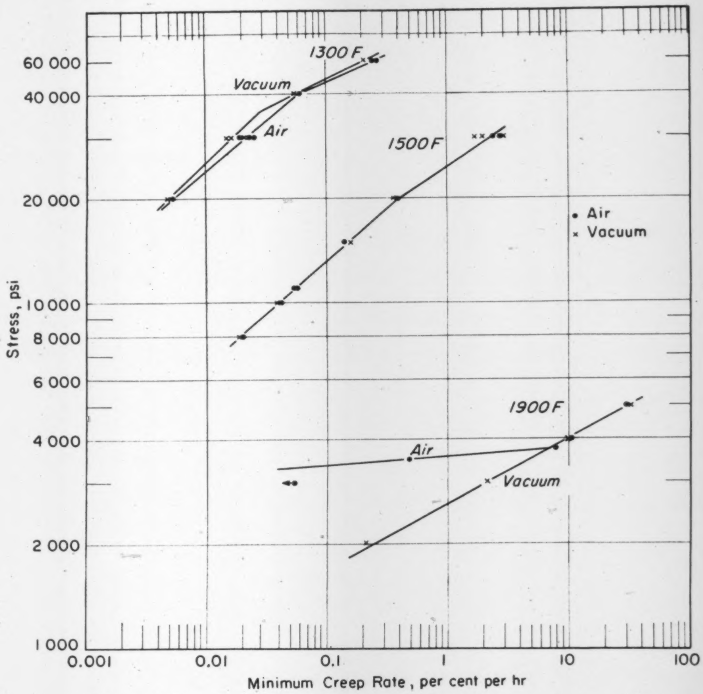

Fig. 2.—Variation of Minimum Creep Rate with Stress in Air and in Vacuum at 1300, 1500, and 1900 F.

peratures and high stresses they are in the air-weakened region.

A prominent feature of the curves is the break or abrupt change in slope of the air curves at 1500 and 1900 F which is not present in the vacuum curves. At the higher temperature the break occurred at a shorter time. Evidently, the change in slope is associated with a marked strengthening in air, which prolongs the time to rupture considerably beyond that expected from extrapolation of short-time data. For example, at 1500 F, the rupture time in air at 8000 psi was 1228 hr while it was 83 hr in vacuum (Fig. 5).

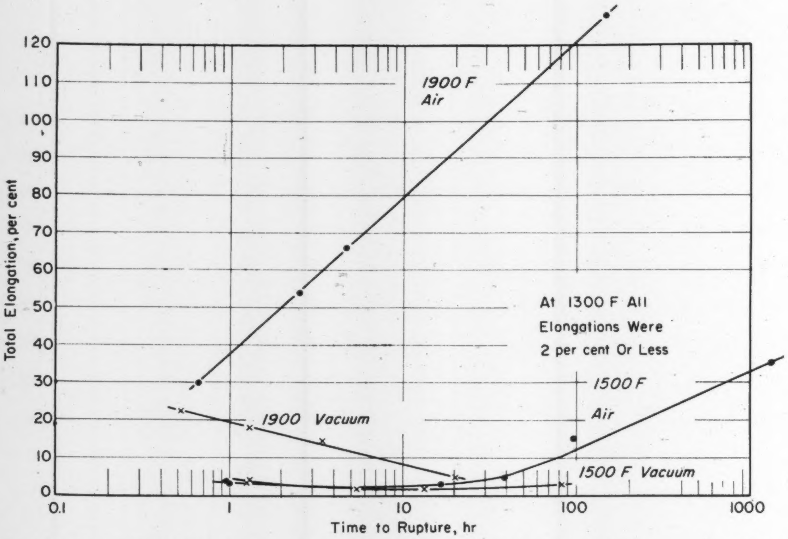

FIG. 3.—Variation of Total Elongation with Rupture Time in Air and in Vacuum at 1500 and 1900 F.

While the rupture times in air and vacuum are different, the minimum creep rates appear to be relatively unaffected by environment but with several exceptions, as shown in Fig. 2. Although the creep rates for the low stress tests at 1900 F are shown to be lower in air, the data do not represent steady-state, second-stage creep because of the unusual shape of the creep curves for these specimens. At 1300 F, even though creep rates at several stress levels appear to be lower in vacuum, the greater amount of scatter in creep rates at this low temperature makes this feature uncertain.

Elongations-at-fracture were low, 4 per cent or less, at 1300 F and at 1500 F except for the long-time air specimens which produced high values that increased with increasing rupture time (Fig. 3). The high elongations were obtained for those specimens whose data points lie to the right of the break in the rupture time curve (Fig. 1). At 1900 F with increasing rupture times, elongation decreased in vacuum but markedly increased in air, yielding a value of 127 per cent for a rupture time of 143.8 hr.

Creep Curves:

These differences in properties of specimens tested in air and vacuum must occur as a result of differences in the creep process in the two environments. Therefore, examination of relative changes in the creep curves should provide information on the operating mechanisms and lead to explanations for the observed behavior. For this purpose, comparisons were made of the curves, which were found to be of the general types shown in Figs. 4 and 5.

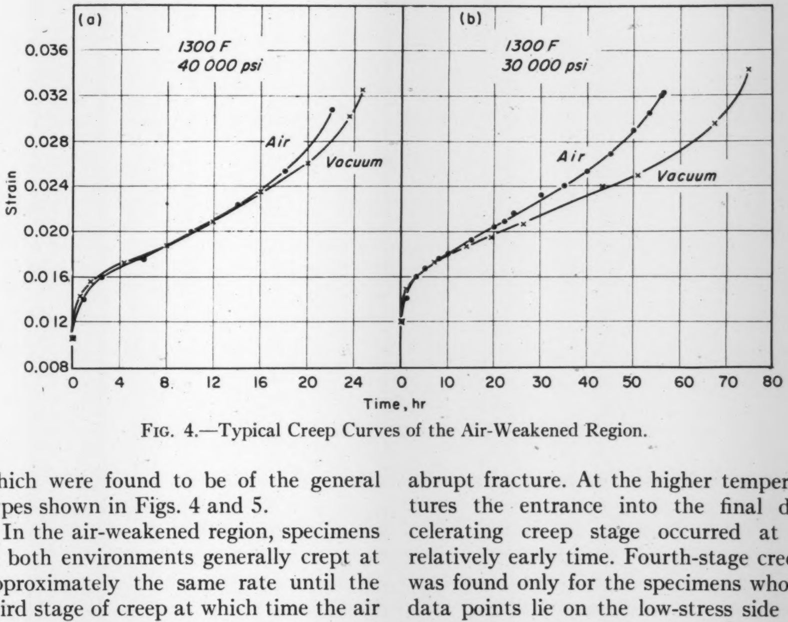

FIG. 4.—Typical Creep Curves of the Air-Weakened Region.

In the air-weakened region, specimens in both environments generally crept at approximately the same rate until the third stage of creep at which time the air specimens began creeping faster, and subsequently failed sooner (Fig. 4(*a*)). Several exceptions were noted, however, of the type shown in Fig. 4(*b*) which revealed a higher creep rate for the air specimen relatively early in the test. Since duplicate tests showed unusually large scatter in the creep rate data, more refined techniques are required to establish the faster creep of the air specimens.

In the air-strengthened region, creep in both environments also progressed at nearly the same rate until shortly prior to fracture at which time the specimen in vacuum began to creep slightly faster. Except when the rupture times were relatively short, the specimen in air, instead of rupturing in the usual third stage of creep, entered a fourth stage of decelerating creep (Fig. 5). The resultant effect was a marked increase in ductility and rupture time and termination in an abrupt fracture. At the higher temperatures the entrance into the final decelerating creep stage occurred at a relatively early time. Fourth-stage creep was found only for the specimens whose data points lie on the low-stress side of the break in the rupture time and creep rate curves (Figs. 1 and 2).

These features of the creep curves enable one to understand the minimum creep rate data in Fig. 2. Since the minimum creep rates of the air specimens were determined from the second stage of creep rather than from the final stages, which in some cases were lower, they were nearly the same as those of the corresponding vacuum specimens over a wide range at 1500 F and at the short times at 1900 F. If the lower creep rate of the final stage, 0.011 per cent per hr, for the 1500 F, 8000 psi air test had been used in preference to the second-stage creep rate of 0.020 per cent per hr, a break would have been indicated in the stress *versus* minimum creep rate curve. The minimum creep rates of the long-time air tests at 1900 F were determined from the last stage of decelerating creep and virtually the only stage, and were much lower than would be expected on the basis of extrapolation. Therefore, a break is shown in the 1900 F curve because there was no conventional second stage from which to measure a steady-state creep rate.

Microstructural Studies:

Metallographic examination of the fractured specimens was performed in an attempt to correlate the effect of environment on creep-rupture properties with its effect on microstructure. In the air-weakened region there were no differences which could be ascribed to environmental effects. It did appear, however, that in this region intergranular cracking occurred predominantly at the specimen surface in short-time tests whereas in long-time tests cracking occurred, to a greater degree, throughout the specimen. Air- and vacuum-tested specimens which were interrupted at various times during the second stage of creep revealed no cracks.

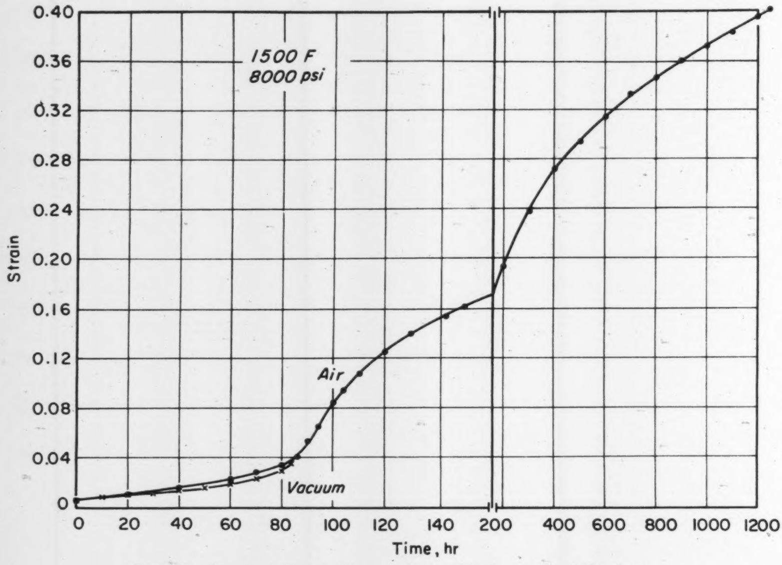

FIG. 5.—Typical Creep Curves of the Air-Strengthed Region.

In contrast to the air-weakened region, profound differences in the microstructures were effected by environment in the air-strengthened region. Although specimens in air contained notably more intergranular cracks than in vacuum, their presence may have been caused by the longer time in test. Large voids were present in the specimens tested for long

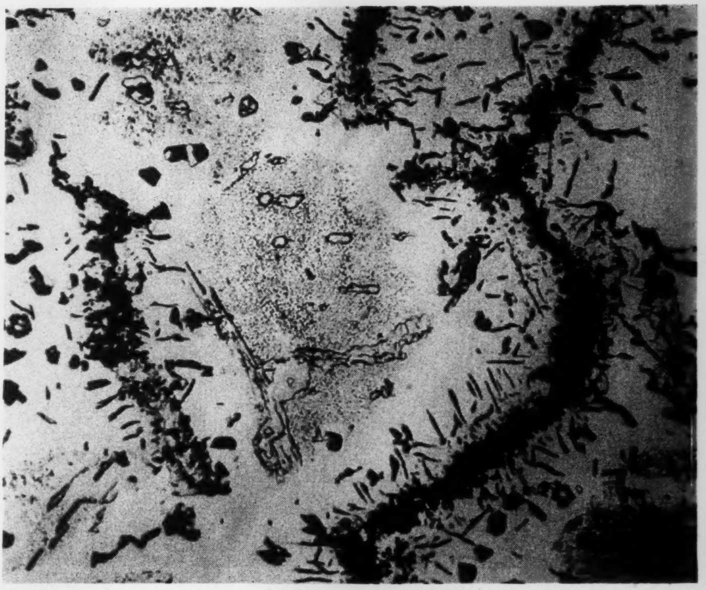

FIG. 6.—Specimen Tested in Air at 1500 F and 8000 psi, 1228 hr to Rupture (× 500).

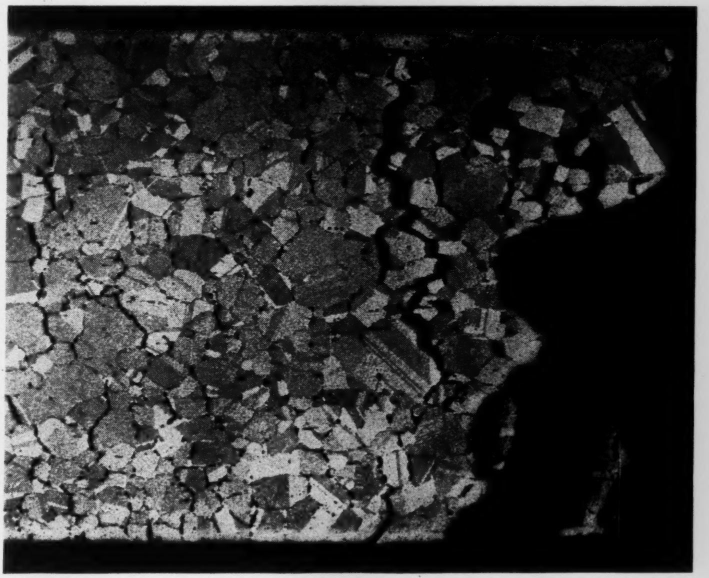

FIG. 7.—Specimen Tested in Vacuum at 1500 F and 8000 psi, 83 hr to Rupture (× 100).

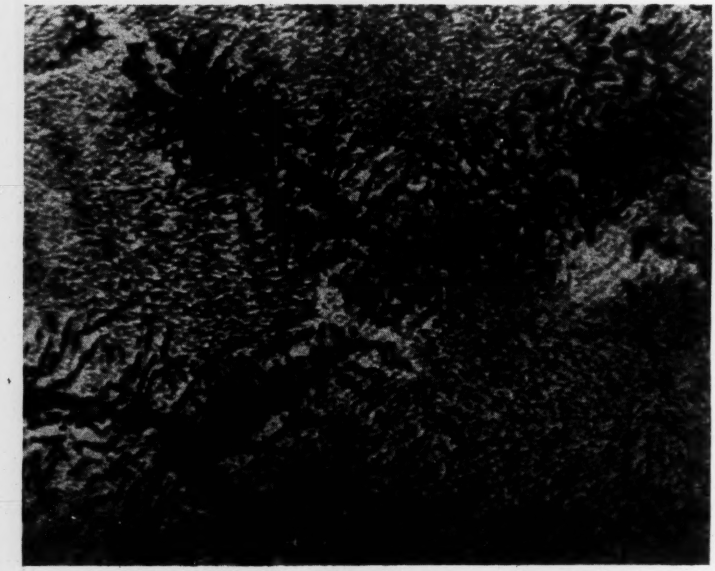

FIG. 8.—Specimen Tested in Air at 1500 F and 8000 psi, Interrupted at 120 hr (× 1500).

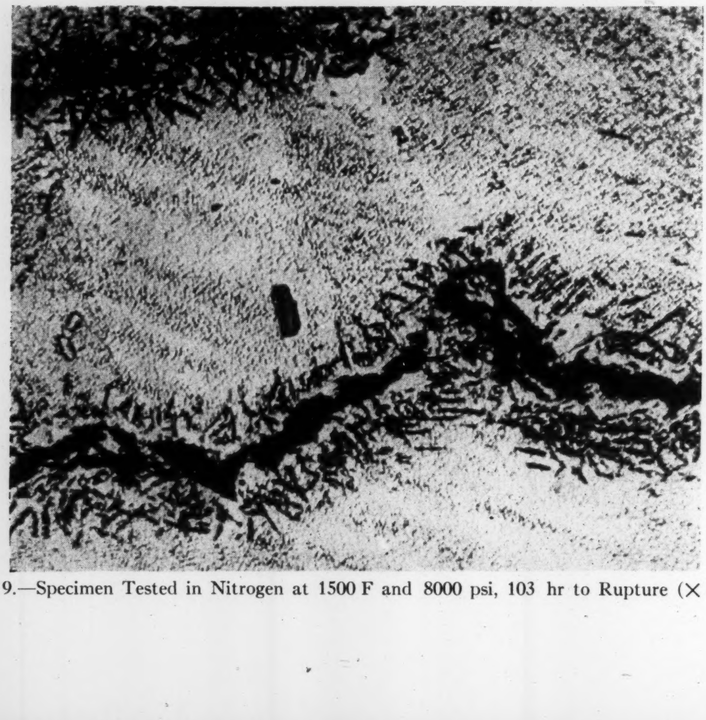

FIG. 9.—Specimen Tested in Nitrogen at 1500 F and 8000 psi, 103 hr to Rupture (× 1500).

times at 1900 F which, in some cases, passed completely through the section.

In those air specimens which lie to the right of the break in the rupture time curve, several new phases had formed principally along the grain boundaries, as shown in Fig. 6. Initially, a needle-like phase formed, and later in the test a polygonal phase appeared. It was not determined if one precipitate was an advanced stage of the other, nor were the phases conclusively identified. There were no new phases in the corresponding vacuum-tested specimens; the only microstructural modification was the presence of fissures (Fig. 7).

After test at 1900 F, small amounts of the γ' phase remaining from the initial heat treatment were observed in the grain boundaries of the vacuum-tested specimens, but none was discerned in the air-tested specimens. This difference may have been caused by depletion of the aluminum by oxide or nitride formation.

In an effort to determine the cause for fourth-stage creep in the low-stress air tests, specimens which were crept at 1500 F and 8000 psi were interrupted at 64 hr, in the early part of the third stage of creep, at 83.5 hr, in order to coincide with rupture of the vacuum-tested specimen, and at 120 hr, after entrance into the final stage of decreasing creep rate (note positions in Fig. 5). Metallographic examination revealed the 64-hr specimen to be essentially free of cracks The one interrupted after 83.5 hr had an amount of cracking of approximately the same order as the specimens which ruptured in vacuum in the same time, while the 120-hr interrupted test displayed considerably more cracking. A small amount of needle-like phase along the grain boundaries was observed in

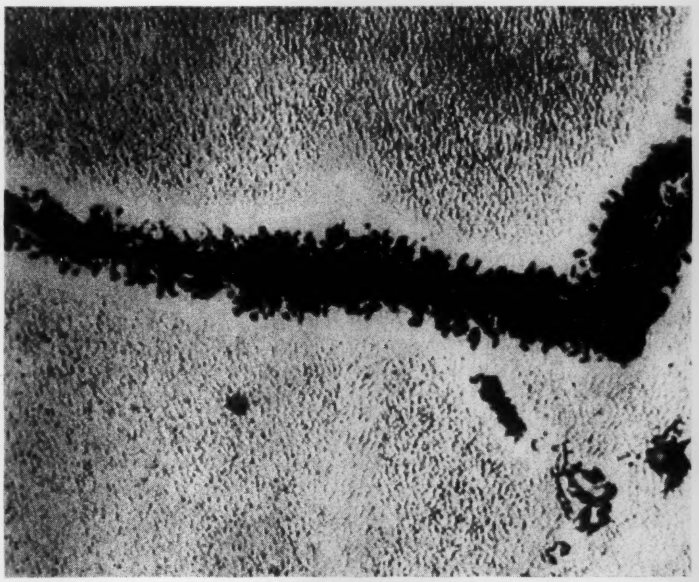

FIG. 10.—Specimen Tested in Oxygen at 1500 F and 8000 psi, 196 hr to Rupture (× 1500).

the 83.5-hr specimen and a much greater amount in the one interrupted after 120 hr (Fig. 8). It should be noted that this new phase was observed only in the air specimens but not in vacuum.

That the needle-like phase is a nitride is indicated by its presence in a specimen tested in nitrogen (Fig. 9) and its absence in one tested in oxygen (Fig. 10). Since this phase was absent in a nickel-chromium alloy (2), it is probably an aluminum nitride. However, this phase evidently is not the major source of strengthening in that the rupture time in nitrogen (Table I) is 103 hr but 196 hr in oxygen. Moreover, the oxygen-tested specimen displayed fourth-stage creep while the one in nitrogen did not.

TABLE I.—CREEP-RUPTURE RESULTS, 1500 F AND 8000 PSI.

Atmosphere	Rupture Time, hr	Minimum Creep Rate, per cent per hr	Elongation, per cent
Air	1228	0.020	35
Vacuum	83.5	0.019	2.8
Oxygen	195.7	0.021	21.2
Nitrogen	102.7	0.019	6.4

It is interesting to note that the needle-like phase did not form in specimens containing fissures (produced by prior vacuum test) on exposure to air for 48 hr at 1500 F without load. Although an influence of stress on the formation is indicated, more detailed experiments are needed to establish the effect.

Gas Content Determinations:

The role of cracking in gas pickup as well as the influence of time and temperature was revealed by gas analyses of fractured specimens (Table II). In air, increases in both oxygen and nitrogen contents occurred at higher temperatures and longer exposure times. After cracking was initiated at the higher temperatures, a large pickup in both oxygen and nitrogen was disclosed. This large increase was probably due to the generation of considerably more surface area through crack formation. Generally it appeared that larger amounts of oxygen were picked up initially, but later in the test the gain in nitrogen was greater. It is possible that this might only be the result of oxides flaking off in the longer-time tests. In vacuum the creation of additional surface area by cracking was seen to facilitate degasification at 1500 F.

TABLE II.—GAS ANALYSES OF SPECIMENS.

Specimen History	Weight, per cent	
	Oxygen	Nitrogen
As-received material	0.016	0.0523
Vacuum annealed material	0.006	0.0389
1300 F, air, 58 hr to rupture	0.011	0.0361
1500 F, air, 64 hr at 8000 psi	0.022	0.0427
1500 F, air, 120 hr at 8000 psi	0.204	0.2133
1500 F, air, 1228 hr to rupture	0.243	0.8094
1500 F, vacuum, 83.5 hr to rupture	0.003	0.0005
1900 F, vacuum, 1.3 hr to rupture	0.014	0.0438
1900 F, air, 2.5 hr to rupture	0.382	0.2699
1900 F, air, 731 hr at 3000 psi	0.739	1.149

At 1900 F, however, a small pickup of gases was found, probably because of the poorer vacuum at this temperature.

DISCUSSION

In order to gain an understanding of the effect of atmosphere on creep and rupture properties, it is essential to examine in detail the creep curves as well as the specimens producing them. Since creep in both air and vacuum was seen to occur, with minor exceptions, at about the same rate during the first and second stages but differed in the third stage, the difference in properties must be related to changes which took place during the third stage when cracks are forming.

In a previous publication (1) a mechanism was proposed involving two competing processes to explain the existence of an air-strengthened and air-weakened region in rupture time and creep rate curves. One of these processes is oxidation strengthening while the other is reduction in strength by the reduced surface energy in air relative to that in vacuum. Reduction of surface energy results in easier crack propagation and consequently faster creep and shorter rupture time. In the air-strengthened region, oxidation is dominant while in the air-weakened region, where conditions are unfavorable for extensive oxidation, the competing process is dominant. Therefore, the process which controls is determined by strain rate and temperature.

Previously (1,2) reversals in both rup-:ure time and minimum creep rate were 'ound, but in the present study, reversals were observed only for rupture time and not for minimum creep rate. In contrast to the more ductile nickel in which crack initiation and growth were observed throughout the second stage of creep, in the present alloy cracking was found to be approximately coincident with entry into the third stage. Therefore, the minimum creep rate was measured for nickel while cracking was taking place, while for this material, it was measured before cracking started. Since environment affects ease of crack propagation, the minimum creep rate is affected by atmosphere for nickel but not for this material. Thus, although the same mechanism of atmosphere effect applies to the two materials, an important difference is that the effect of environment is observed in a later stage.

By means of this mechanism it is now possible to understand the form of the creep curves. In the air-weakened region (Fig. 4(*a*)), the air and vacuum specimens elongate at the same rate until cracking is initiated, at which time the reduced surface energy in air results in accelerated propagation and failure.

In the air-strengthened region, since cracking does not lead to immediate failure, as shown in Fig. 5, the rupture time is prolonged. The marked strengthening which occurred in the long-time, low-stress tests (conditions conducive to greater oxidation) was found to be related to the oxygen in the air environment more so than the nitrogen. This strengthening process became effective after fissures were formed in the third stage of creep, as indicated by the large pickup in gases at this point. While badly fissured, the specimen, instead of rupturing in the third stage, entered a final stage of decelerating creep, thereby yielding an unusual type of creep curve.

Although the exact mechanism by which this marked strengthening occurs is not known, it appears to be related to extensive oxidation. Three possible ways have been considered in which oxidation may affect cracking and resultant strength changes.

1. Oxidation ahead of the crack may strengthen the alloy.
2. Oxidation may blunt the tip of a crack and thus reduce the stress concentration there.
3. Oxidation of the fracture surfaces can bridge the crack and measurably increase the load bearing area.

In view of the observation that a specimen which failed in vacuum in 83.5 hr and one which was interrupted in air at the same time had approximately the same amount of intergranular cracking, it appears that mechanism 3 acts to prevent failure, even though cracking is well advanced.

Another indication of the influence of oxidation is the positions in Fig. 1 of the reversal points and of the breaks in the rupture time curve in air. These features were observed to occur at shorter times relative to those for a nickel-chromium alloy (2). Since these materials have comparable oxidation resistance in the temperature range studied (5,6), it appears that this displacement to shorter time may be attributed to the greater surface-to-volume ratio for these thinner specimens.

SUMMARY

The significant findings of the investigation are as follows:

1. At low temperatures and high stresses time-to-rupture was longer in vacuum than in air, but at high temperatures and low stresses the reverse was true. Conventional minimum creep rates were generally unaffected by environment.
2. The observed behavior is in agreement with previously proposed mechanisms involving reduction of strength due to lowered surface energy and strengthening by oxidation.
3. In this brittle alloy, the effect of environment was not observed until later in the test in comparison with more ductile materials.
4. An unusual creep curve was obtained in air at the higher temperatures. The specimen instead of rupturing in the normal third stage of creep entered a fourth stage of decelerating creep which delayed rupture.
5. Ductility was not significantly altered by environment at low temperatures. However, at high temperatures with increasing rupture time, total elongation increased markedly in air, but decreased in vacuum.

Acknowledgment:

The experimental program was conducted by J. T. Atwell. This program was supported by Bureau of Aeronautics, Code NP.

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DISCUSSION

MR. E. N. SKINNER.[1]—This excellent and interesting investigation calls attention to an important and perhaps little appreciated fact: air, the universal environment for high-temperature mechanical testing is far from inert and by its presence or absence may exert a profound influence upon the strength properties of heat-tolerant metals and alloys. We and others have observed that the creep-rupture testing in air at a sufficiently high temperature of materials of this type may lead to erroneous impressions as to the actual strength because of the benefit resulting from the formation of oxide films on the surface or of oxide and nitride precipitation within the structure. This effect is, of course, masked or not found in alloys that form nonprotective oxides where spalling results in a continual loss of load-carrying sections. However, in alloys featuring the high order of oxidation resistance typical of the nickel-base chromium types, the strengthening may be so pronounced as to suggest that it is futile to test specimens in the form of strip (or small diameter rods) at high temperatures and low stresses such that the test duration is sufficiently long to introduce complications due to oxidation.

The magnitude of oxidation strengthening is often remarkable as in one case where we have tested in creep rupture a round bar specimen of Inconel (78 nickel - 15 chromium) at 2100 F and 850 psi. The amount of elongation was very large but just at a time when it appeared that rupture was imminent, the rate of third stage extension commenced to decrease not unlike the authors' specimen which exhibited a "fourth" stage of creep. Ultimately, the elongation of the Inconel bar attained a zero rate and so remained for more than 1000 hr, after which the test was discontinued. Examination of the specimen which had no ductility whatsoever indicated that the usual network of rupture cracks had become filled with oxide, which eventually became the continuous phase and thereafter supported the load.

MR. PAUL SHAHINIAN (*author*).—I agree with Mr. Skinner's remarks. There is no question that in environmental testing the ratio of surface area to volume is very important. The greater atmosphere effect expected from high ratio values, of course, is one reason why we used sheet materials. As to the actual use of such data I believe there are applications for this and similar materials in sheet form, for example, in cladding.

MR. J. R. LEWIS[2] (*by letter*).—The effect of atmosphere upon the mechanical properties of materials has been of extreme interest to us for some time. The excellent paper presented by the authors is further verification that in selecting materials operating media must be as carefully considered as are operating stress or temperature.

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[1] Development and Research Division, The International Nickel Co., Inc., New York, N. Y.

[2] General Electric Co., Aircraft Nuclear Propulsion Department, Cincinnati, Ohio.

The authors' findings relating to internal oxidation and nitride formation in nickel-chromium-aluminum alloys are in agreement with investigations at our laboratories. We have also found that nitrides will form in type 310 stainless steel, in 80 nickel-20 chromium material and in nickel-chromium-columbium-silicon alloys upon exposure to air at elevated temperatures.

It is interesting to note that the stress-rupture life in air is over six times that in oxygen alone and of an order of magnitude greater than the life in nitrogen. From this one would infer the cumulative effect of nitrogen plus oxygen to be due perhaps to the formation of oxinitrides which are more effective strengtheners or to a change in the nature or distribution of the oxide and nitride phases. We would like to ask the authors whether any evidence exists which could explain the marked difference in behavior in air as opposed to nitrogen and oxygen.

MR. SHAHINIAN.—In regard to Mr. Lewis's query, we do not have evidence to support an explanation for the longer rupture life in air compared to that in oxygen or nitrogen. It could be due to the effects suggested by him, or possibly to a critical oxidation rate required for optimum strength, or to the minor components of air. Similar differences have also been observed with other materials.

MECHANICAL AND PHYSICAL PROPERTIES OF THREE LOW-SHRINKAGE, COPPER-BASE CASTING ALLOYS*

By J. G. Kura[1] and R. M. Lang[2]

Synopsis

Accurate measurements were made for the mechanical and physical properties of 80-10-10, 85-5-5-5, and Navy "M" alloys at various temperatures ranging from −40 to 550 F. The properties measured were ultimate strength, yield strength, elongation, reduction of area, Young's modulus, compressive strength, V-notch Charpy impact strength, Brinell hardness, fatigue, machinability, melting range, patternmaker's shrinkage, density and specific gravity, electrical resistivity, thermal conductivity, and thermal expansion. Many of the data were previously nonexistent.

A program was sponsored by the Brass and Bronze Ingot Inst. to provide accurate data for various mechanical and physical properties of sound test bars from good-quality melts of three copper-base casting alloys. These ingot alloys are designated as 2A, 3A, and 4A by the ASTM Specification B 30 – 54,[3] and the trade designations are Navy "M", 80-10-10, and 85-5-5-5, respectively. The casting specifications are ASTM B 143 – 52,[4] 2A; B 144 – 52,[5] 3A; and B 62 – 52,[6] respectively.

Although data on some of the mechanical and physical properties of these alloys are available, the authenticity of these data is not precisely known. For example, little is known about the exact composition, quality of the melts, and soundness of the test bars used in determining the properties listed in the various reference sources. Authoritative data on the elevated-temperature properties of these alloys are practically nonexistent.

Pouring temperature has a marked effect on the mechanical properties of castings. Therefore, the test-bar castings for this program were poured within a predetermined range of temperature to produce optimum tensile properties.

Data were obtained on 16 mechanical and physical properties. Some of these properties were measured at room temperature, but most of them were measured at −40, 75, 250, and 450 or 550 F. The specific properties and the methods used to measure them are presented in separate sections of the paper.

An important part of the program dealt with the determination of the creep

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* Presented at the Sixty-first Annual Meeting of the Society, June 22–27, 1958.

[1] Chief, Nonferrous Metallurgy Division, Battelle Memorial Institute, Columbus, Ohio.

[2] Formerly Principal Metallurgist, Battelle Memorial Institute; presently Metallurgist, Engineering Design Division, Douglas Aircraft Co., El Segundo, Calif.

[3] Specification for Copper-Base Alloys in Ingot Form for Sand Castings (B 30 – 54), 1955 Book of ASTM Standards, Part 2, p. 75.

[4] Specification for Tin-Bronze and Leaded Tin-Bronze Sand Castings (B 143 – 52), 1955 Book of ASTM Standards, Part 2, p. 229.

[5] Specification for High-Leaded Tin-Bronze Sand Castings (B 144 – 52), 1955 Book of ASTM Standards, Part 2, p. 233.

[6] Specification for Composition Brass or Ounce Metal Castings (B 62 – 52), 1955 Book of ASTM Standards, Part 2, p. 119.

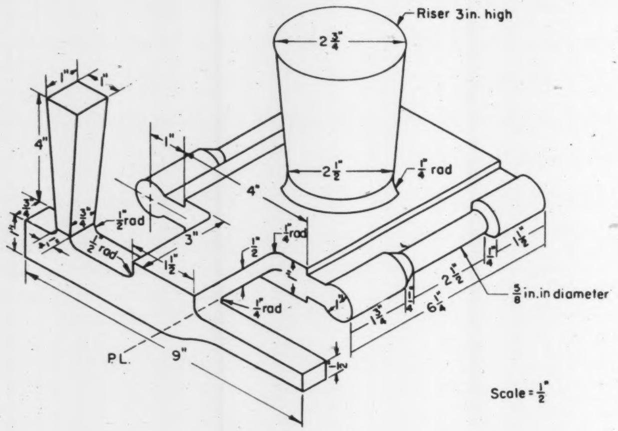
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FIG. 1.—Double Horizontal Full-Web Test Bars, Similar to Fig. 3 of ASTM Recommended Practice B 208 – 54[8] Except for Modifications in the Gating System.

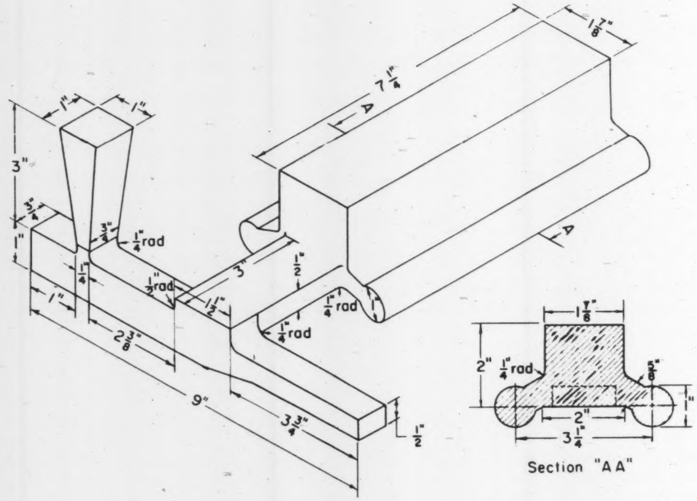
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FIG. 2.—Modified 1-in. Web-Webbert Test-Bar Casting.

properties. Those results are presented in a companion paper.[7]

The data presented in this paper constitute the first portion of a continuing program in which additional alloys will be studied.

EXPERIMENTAL PROCEDURE

Preparation of Specimens:

All of the specimens for this program were cast in green sand molds in accordance with Am. Foundrymen's Soc. (AFS) recommendations except the specimens for determining the patternmaker's shrinkage. The latter were cast in baked sand molds.

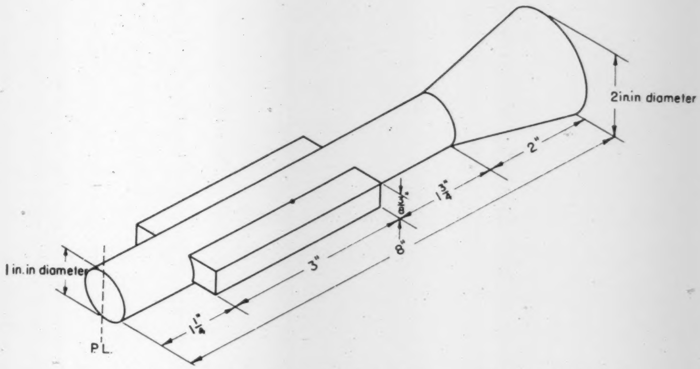

FIG. 3.—Casting Used for Measuring Patternmaker's Shrinkage.

The casting designs used to provide specimens for all the different tests were as follows:

1. Double Horizontal Full-Web type, Fig. 1 (ASTM Recommended Practice B 208 - 54,[8] Fig. 3)
2. Modified 1-in. Web-Webbert, Fig. 2
3. Patternmaker's shrinkage bar, Fig. 3.

The Double Horizontal Full-Web test bar was used because it is one of the designs specified by ASTM for low-shrinkage alloys. Also, previous research had shown that this type of casting was the most satisfactory design for the 85-5-5-5 alloy (**1**)[9]. However, the gating system was modified to conform to more recent principles of gating (**2**). This test-bar design was used for 80-10-10 and Navy "M" alloys because these alloys have casting characteristics similar to those of the 85-5-5-5 alloy.

When the desired size of the specimens was too large for them to be machined from the Double Horizontal Full-Web bar, the Modified 1-in. Web-Webbert test bar was used. The Double Horizontal Full-Web test bars were used to determine the tensile properties, hardness, melting range, and density. The special bar shown in Fig. 3 was used to measure patternmaker's shrinkage. All of the other properties were measured from the Modified 1-in. Web-Webbert test bars.

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[7] Ward F. Simmons and J. G. Kura, "The Creep Properties of Three Low-Shrinkage, Cooper-Base Casting Alloys," p. 791, this publication.

[8] Recommended Practice for Tension Test Specimens for Copper-Base Alloys for Sand Castings (B 208 - 54), 1955 Book of ASTM Standards, Part 2, p. 345.

[9] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 790.

To determine whether the tensile properties of the Modified 1-in. bar were comparable with those of the Double Horizontal bar, both types were cast from the same melts made in duplicate. The results are shown in Table I and indicate that the tensile properties of the Modified 1-in. bars were lower than those of the Double Horizontal bars. This was expected, since, in general, castings of larger section thickness have lower tensile properties than castings of smaller section thickness. The reduction in properties is attributed to the larger grain size and more open structure that result from the lower cooling rate of the larger castings. In general, the difference in tensile properties of these two types of castings was small. Therefore, the Modified 1-in. bars were considered to be acceptable for the type of evaluation for which they were to be used, especially since the Modified 1-in. bars were radiographically sound.

TABLE I.—EFFECT OF CASTING DESIGN ON ROOM-TEMPERATURE TENSILE PROPERTIES.

Design	Pouring Temperature, deg Fahr	Tensile Strength, psi			Elongation, per cent		
		Maximum	Minimum	Average^{b}	Maximum	Minimum	Average^{ab}
80-10-10-ALLOY							
Double Horizontal Full-Web	2075	38 400	37 400	38 000^{4}	31	29	30^{4}
Modified 1-in. Web-Webbert	2090	34 800	34 300	34 600^{2}	26	24	25^{2}
85-5-5-5 ALLOY							
Double Horizontal Full-Web	2160	35 000	35 000	35 000^{2}	35	33	34^{2}
Modified 1-in. Web-Webbert	2135	32 800	31 800	32 300^{4}	33	30	31^{4}
NAVY "M" ALLOY							
Double Horizontal Full-Web	2150	41 700	40 000	40 800^{4}	52	46	48^{4}
Modified 1-in. Web-Webbert	2140	36 900	36 200	36 500^{4}	42	38	40^{4}

[a] The standard deviation for tensile strength is ± 300 psi; that for elongation, ± 1 per cent. (See section on "Reliability of Data" for method used to calculate the standard deviation.)

[b] Superscript numerals indicate the number of specimens.

Melting:

Even when considerable care is exercised in the melting of brass and bronze alloys, the melts may occasionally absorb a large amount of gas, which causes a marked reduction in the tensile properties of the castings. Therefore, to determine the melt quality, tension tests were made on castings from each melt before the melt was considered acceptable.

Previous work (1) had shown that the Double Horizontal Full-Web test-bar castings poured at about 2150 F were more sensitive to variations in melt quality of the 85-5-5-5 alloy than any of many other types of test-bar castings. Because the 80-10-10 and Navy "M" alloys had casting characteristics similar to those of the 85-5-5-5 alloy, the same procedure was followed in evaluating their melt quality as for the 85-5-5-5 alloy.

To determine the pouring temperature that would result in optimum tensile properties, melts of the 80-10-10 and Navy "M" alloy were made in duplicate,

TABLE II.—AVERAGE CHEMICAL ANALYSIS FOR CASTINGS FROM EACH ALLOY TYPE.[a]

Element	Chemical Analysis, per cent		
	80-10-10	85-5-5-5	Navy "M"
Copper	79	85	88
Tin	9.3	4.4	5.7
Lead	10.2	5.1	1.4
Zinc	0.27	4.6	4.4
Iron	0.01	0.11	0.07
Antimony	0.26	0.17	0.13
Nickel	0.43	0.7	0.6
Phosphorus	0.01	0.01	0.01

[a] Eleven heats of 80-10-10 alloy, seven of 85-5-5-5 alloy, and nine of Navy "M" alloy.

and the Double Horizontal Full-Web castings were poured at five different temperatures from each melt. Tensile data were then obtained for each casting. From these data, the pouring temperatures selected were 2050 F for the 80-10-10 alloy and 2150 F for the Navy "M" alloy.

All of the alloys were melted in a silica-lined, high-frequency induction furnace without a cover over the melt. The melts consisted of ingot that met the ASTM specifications for composition.[3] The number of castings poured from each melt was limited to six or seven, so that all castings could be poured close to the preestablished temperature. The shake-out time for all castings throughout the program was ½ hr.

The average analyses for the compositions of the various melts used in the program are presented for each type of alloy in Table II. All of the melts were within the composition range specified by the ASTM.[3]

The results of the melt-quality tests conducted on specimens machined from the Double Horizontal Full-Web test-bar castings are summarized in Table III. In all cases, the tensile properties of the casting exceeded the minimum set by ASTM specifications.

In addition to checking the composition and quality of each melt, specimens were radiographed before they were subjected to long-time tests such as creep and fatigue.

TABLE III.—SUMMARY OF THE ROOM-TEMPERATURE TENSILE PROPERTIES DETERMINED FOR MELT-QUALITY EVALUATION OF ALL OF THE HEATS.

Alloy	Tensile Strength, psi			Elongation in 2 in., per cent		
	Maximum	Minimum	Average[a,b]	Maximum	Minimum	Average[a,b]
80-10-10	41 800	36 500	39 200[18]	34	26	30[18]
85-5-5-5	37 500	33 100	35 300[14]	40	27	32[14]
Navy "M"	41 600	39 700	40 700[12]	53	40	45[12]

[a] Standard deviation for tensile strength is ±300 psi; that for elongation, ±1 per cent.
[b] Superscript numerals indicate the number of specimens.

Reliability of Data:

In the evaluation of the three alloys, it was possible to treat statistically all of the individual values for all of the alloys simultaneously for a specific property (3). This was done in the present program to arrive at the standard deviation of $\pm 1\ \sigma$ for each average value listed in the various tables. The value for the standard deviation of $\pm 1\ \sigma$ indicates the limits that would encompass two thirds of all additional experimental results that might be obtained in the future. Thus, the smaller the value for the standard deviation, the greater the reliability of the data. In each table where data are averaged, the value for the standard deviation is given in the footnotes.

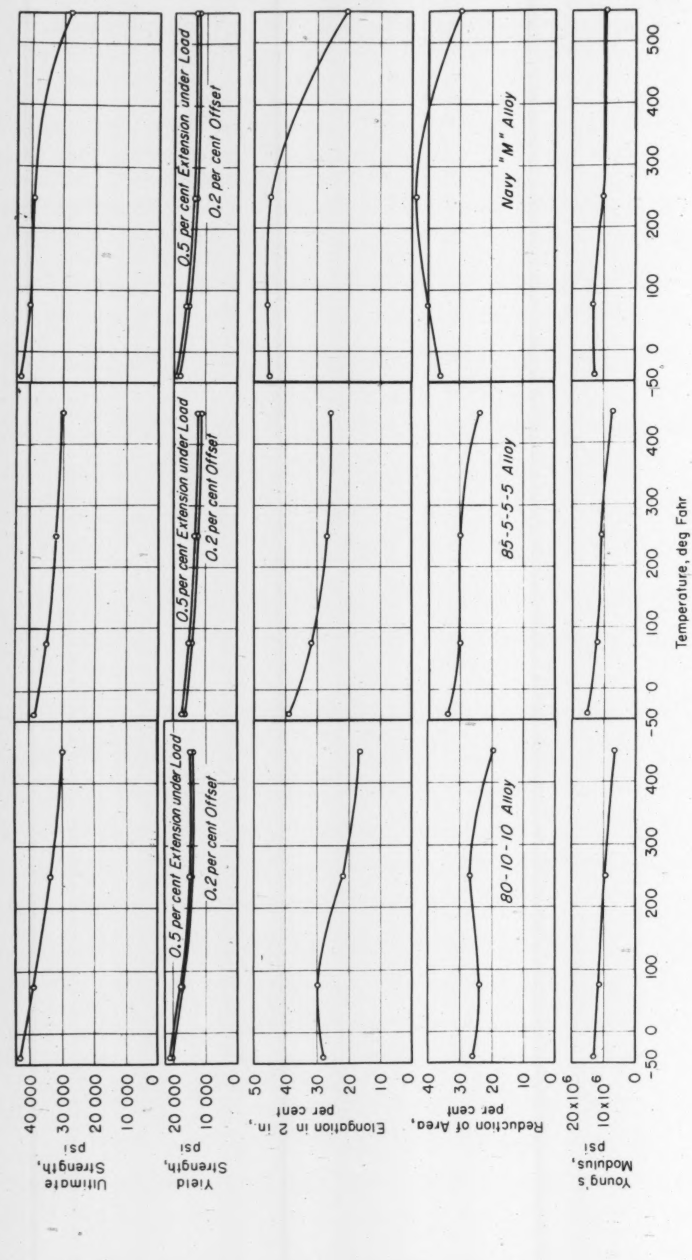

FIG. 4.—Tensile Properties.

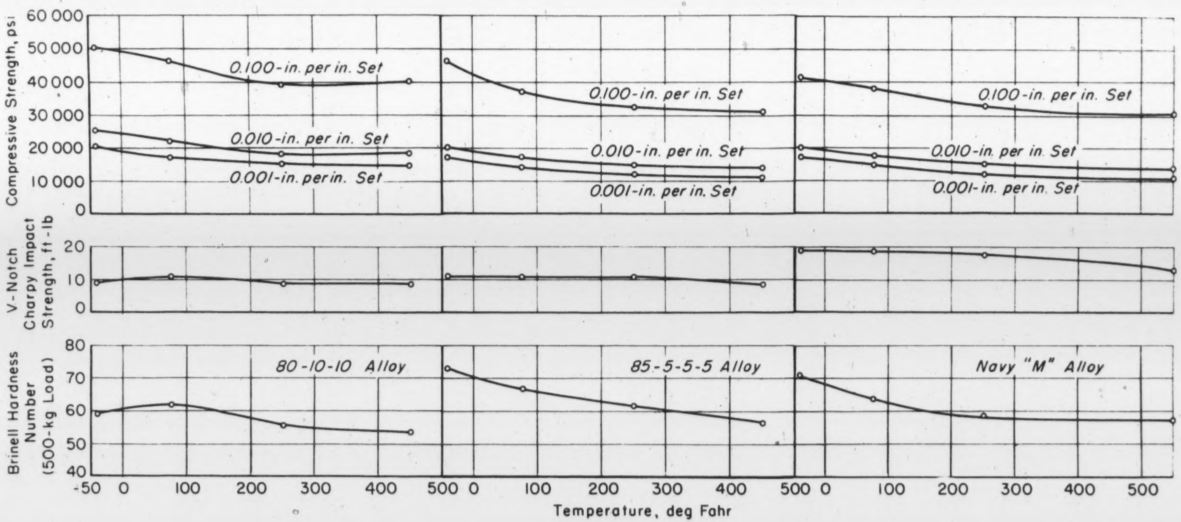

FIG. 5.—Compressive Strength, V-Notch Charpy Impact Strength, and Hardness.

DATA ON PROPERTIES

Tensile Properties:

The tensile properties of the three alloys were measured on bars in duplicate at −40 F, room temperature, 250, and 450 or 550 F. Data obtained included (*a*) yield strength at both 0.2 per cent offset and at 0.5 per cent extension under load, (*b*) ultimate strength, (*c*) elongation, (*d*) reduction of area, and (*e*) modulus of elasticity. The tension specimens conformed in detail to Fig. 5 of ASTM Recommended Practice B 208 – 54.[8]

Hydraulic testing machines were used which are accurate to ±0.5 per cent. The rate of straining was 0.002 in. per in. per min below the yield strength and 0.05 in. per in. per min above the yield strength. The specimens were held at the testing temperature to within ±2 F for at least 20 min prior to load application.

The strain on the tension specimens tested at −40 F and at room temperature was determined by means of two electric strain gages attached in series for each specimen. The sensitivity of the strain gages was 0.000002 in. per in. of gage length. For the specimens tested at elevated temperature, the strain was measured with an extensometer over a 2-in. gage length. The sensitivity of the extensometer was 0.00003 in. per in. of gage length.

Figure 4 shows the tensile properties that were obtained at the various temperature levels. The accuracy of the individual values is estimated to be as follows:

Property	Temperature, deg Fahr	Experimental Error	Standard Deviation
Ultimate strength[a]	-40 to 550	±1 % of the ultimate strength	±300 psi at 75 F, ±750 psi at other temperatures
Yield strength	-40 to 75	±1 % of the yield strength	±170 psi
Yield strength	250 to 550	±2 % of the yield strength	±170 psi
Reduction of area	-40 to 550	±1½ %	±2 per cent
Elongation[a]	-40 to 75	±1 %	±1 per cent at 75 F, ±2 per cent at other temperatures
Young's modulus	-40 to 75	±2 % of the modulus	±430 000 psi
Young's modulus	250 to 550	±4 % of the modulus	±430 000 psi

[a] Twenty-three specimens were tested at room temperature for the 80-10-10 alloy, 18 for the 85-5-5-5 alloy, and 18 for the Navy "M" alloy. Specimens in duplicate were tested at all other temperatures and for the other properties of the three alloys.

The data in Fig. 4 show that the strength, ductility, and modulus of elasticity of all the alloys generally decreased with increasing temperature. At the maximum temperature, the Navy "M" alloy retained about 70 per cent of its room-temperature strength, and the 80-10-10 and 85-5-5-5 alloys retained about 75 and 85 per cent of this room-temperature strength, respectively.

Compressive Strength:

The compressive strength of the three alloys was measured at various temperatures on cylindrical specimens $\frac{3}{4}$ in. in diameter and 2 in. long. The specimens were machined to ±0.0005 in. on the diameter and ±0.005 in. on the length. The opposite ends of each specimen were ground to make them parallel to within ±0.0002 in.

The rate of straining with hydraulic testing machines was 0.001 in. per in. per min below the 0.1 per cent offset yield strength and 0.02 in. per in. per min above the 0.1 per cent offset yield strength. The sensitivity of the extensometer was 0.00003 in. per in. of gage length. The compressive strength was measured from stress-strain diagrams at 0.1, 1.0, and 10 per cent offset, which corresponds to permanent sets of 0.001, 0.010, and 0.100 in., respectively. Before load application, the specimens were held at the testing temperature to within ±5 F for at least 20 min.

According to ASTM Methods E 9 – 52 T,[10] the strain on compression specimens is to be measured over a gage length not closer to the ends of the specimen than an amount equal to one quarter of the diameter of the specimens. In the present program, an arrangement was used to provide (*a*) high sensitivity for determining the elastic modulus for establishing the slope of the offset lines, and (*b*) sufficient range for determining the plastic compression to the desired maximum of 10 per cent offset. Set screws in the attachment rings of the extensometer contacted the specimen $\frac{1}{4}$ in. from each end, and the rings contacted the ram and base of the compression machine. This achieved alignment of the specimen and stability of the extensometer. If the extensometer ring clamps were located farther from the ends of the specimen, alignment would have been a problem at the various test temperatures and the set screws would have restricted expansion of the specimen as it approached 10 per cent compression. Therefore, in this arrangement, the strain was measured over the full 2-in. length of the specimen.

Figure 5 gives the compressive strengths that were obtained at the variout temperatures. They show that there was first a rapid and then a gradual decrease in the compressive strength for each of the alloys as the temperature was increased from −40 to 450 or 550 F. The accuracy of these data is estimated to be as follows:

Compressive Strength[a]	Experimental Error, per cent	Standard Deviation
At 0.001-in.-per-in. permanent set......	±1	600 psi
At 0.010-in.-per-in. permanent set......	±1	
At 0.100-in.-per-in. permanent set......	±2	

[a] Specimens in duplicate were tested at each temperature level except for the 80-10-10 alloy, which was tested in quadruplicate at 250 and 450 F.

Impact Strength:

Impact strength was determined on specimens in triplicate at various temperatures from −40 F to 450 or 550 F. Standard V-notch Charpy specimens were used; the design conformed to Fig. 3 in ASTM Methods E 23 – 56 T.[11] The pendulum had an energy of 110 ft-lb at the moment of impact and was moving at a velocity of 12.75 ft per sec.

Figure 5 shows the strengths of the notched bars at various temperature levels. The impact strength was fairly constant for the 80-10-10 and 85-5-5-5 alloy over the range of test temperatures and dropped from 19 ft-lb at −40 F to 14 ft-lb at 550 F for the Navy "M" alloy. These data are estimated to be accurate to within $\pm\frac{1}{2}$ ft-lb, and the standard deviation for the average values is ±0.6 ft-lb.

Hardness:

Brinell hardness was determined on flat surfaces machined on the side of the grip ends of bars that had been used to determine room-temperature tensile properties. All of the impressions were made with a 10-mm-diameter tungsten carbide ball, and a load of 500 kg was applied for 30 sec. Impressions were made after the specimen had been in the bath for 30 min. Three impressions were made on each specimen and two specimens of each alloy were tested at −40, 75, 250, and 450 or 550 F.

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[10] Tentative Methods of Compression Testing of Metallic Materials (E 9 – 52 T), 1955 Book of ASTM Standards, Part 1, p. 1566; Part 2, p. 1261.

[11] Tentative Methods of Notched Bar Impact Testing of Metallic Materials (E 23 – 56 T), 1956 Supplement to Book of ASTM Standards, Part 1, p. 351; 1957 Supplement to Book of ASTM Standards, Part 2, p. 337.

Fatigue Strength:

Fatigue studies were conducted on specimens of the design shown in Fig. 6 After polishing, the average surface finish was 5 microinches rms. R. R. Moore rotating-beam machines were used. The specimens were loaded as a simple beam while rotated at a speed of 10,000 rpm.

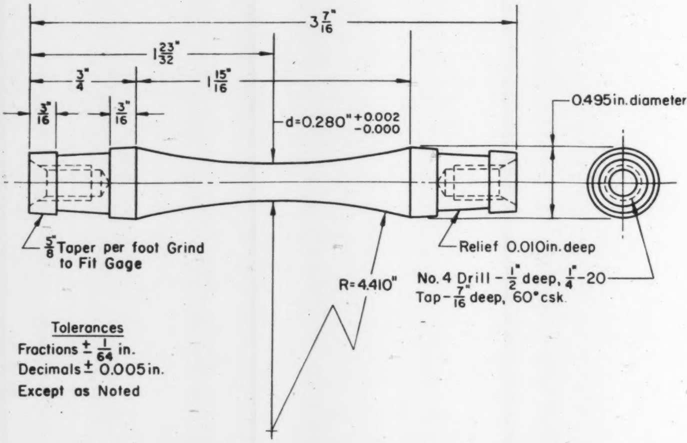

Fig. 6.—R. R. Moore Rotating-Beam Fatigue Specimen.

Figure 5 shows the hardness of each alloy at the various temperatures. The accuracy of the individual measurements is estimated to be ±1 per cent, and the standard deviation of the average values is ±2 Brinell hardness number.

As would be expected, a decrease in hardness occurs for each alloy as the temperature is increased. The hardness of the 80-10-10 alloy dropped about 5 points to 54 Brinell at 450 F. The 85-5-5-5 and Navy "M" alloy were quite similar in behavior. Their hardnesses were slightly above 70 Brinell at −40 F and dropped to only slightly below 60 Brinell at the highest test temperature.

The data are plotted in Fig. 7. The solid curves, representing the mean value and limiting range, were determined as follows. In the finite-lifetime region (in general, less than 10^7 cycles), a statistical analysis was made at each stress level for which data were available for four or more specimens. The mean value for the lifetime at each of these stress levels assisted in locating the solid line in the figures. The broken lines in the figures were arrived at in a similar manner. With reference to statistical terminology, the broken lines represent a standard deviation of $\pm 1\ \sigma$.

From the solid line representing the mean values in each figure, the mean fatigue limit for the three alloys at 100

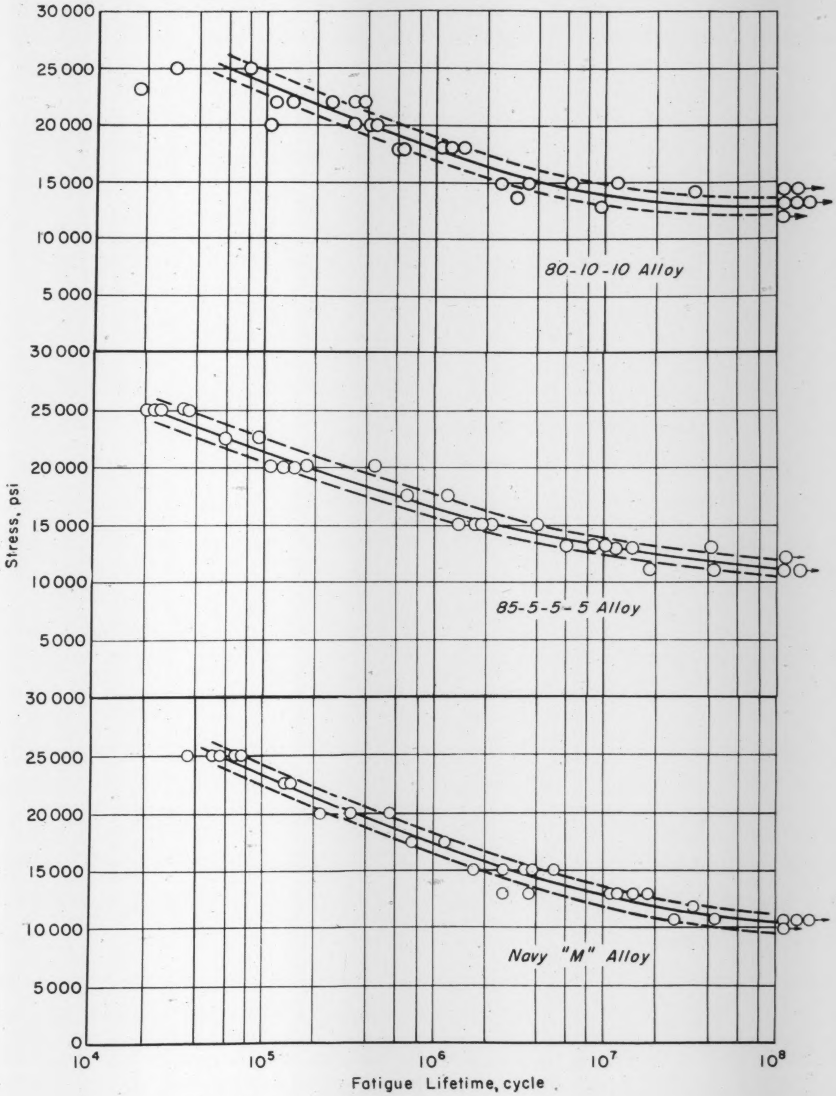

FIG. 7.—Fatigue Characteristics.

million cycles was estimated to be as follows:

Alloy	Mean Fatigue Limit, psi
80-10-10	13 000
85-5-5-5	11 000
Navy "M"	11 000

Machinability:

Machinability was determined on a lathe in which a constant thrust is applied to the tool carriage by a weight-and-pulley system. The machinability ratings are based on the number of spindle revolutions required for a tool travel of 0.1 in. A metal of good machinability will require fewer revolutions of the spindle for a given amount of tool travel than will a metal of poor machinability.

TABLE IV.—MACHINABILITY RATINGS.

Alloy	Machinability Rating Index
Free-cutting brass[a]	100
80-10-10	97
85-5-5-5	84
Navy "M"	42

[a] Nominal composition of the wrought bar stock was 61.5 per cent copper, 35.5 zinc, 3 lead (ASTM Specification B 16 - 52).[12]

TABLE V.—MELTING-POINT DATA.

Alloy	Beginning of Melting, deg Fahr						End of Melting, deg Fahr		
	Lead Phase			Copper Phase					
	Maximum	Minimum	Average[a,b]	Maximum	Minimum	Average[a,b]	Maximum	Minimum	Average[a,b]
80-10-10	598	598	598^3	1410	1400	1403^3	1708	1702	1705^3
85-5-5-5	602	600	601^2	1575	1560	1568^3	1850	1846	1849^3
Navy "M"	...	...	...	1520	1515	1518^2	1816	1803	1810^2

[a] Standard deviation for the average values is ±5 F.
[b] Superscript numerals indicate the number of specimens.

The procedure consisted of making a $\frac{1}{8}$-in. turning cut at a mean speed of 75 surface ft per min on bars measuring $\frac{7}{8}$ in. in diameter by 7 in. in length. Wrought bar stock of free-cutting brass was used as a standard for comparison purposes.

The machinability ratings based on tests conducted in quadruplicate are presented in Table IV. The data show that, of the three alloys, the 80-10-10 had the highest machinability and the Navy "M" alloy had the lowest. The accuracy of the machinability test is estimated to be ±5 per cent with respect to the standard reference alloy.

Melting Range:

The melting range of each of the three alloys was determined by the differential thermal analysis technique, which is one of the most accurate methods available. This technique consists of recording the temperature of the specimen as well as the temperature difference between the specimen and a neutral body. Copper rod was used as the neutral body. The alloy specimens as well as the copper bars were $\frac{1}{2}$ in. in diameter and $\frac{3}{4}$ in. long.

With this technique of thermal analysis, it was possible to determine the temperature at which the lead-rich phase began to melt in the 80-10-10 and 85-5-5-5 alloys. However, with the Navy "M" alloy, melting of the lead-rich phase could not be detected. Apparently the lead content of the Navy "M" was too low to cause an appreciable arrest in the heating curve.

The data for the melting ranges of the three different alloys are presented in

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[12] Specification for Free-Cutting Brass Rod, Bar, and Shapes for Use in Screw Machines (B 16 - 52), 1955 Book of ASTM Standards, Part 2, p. 54.

Table V and are estimated to be accurate to within ±5 F. These data, which were obtained by heating the specimens, are regarded as a more reliable indication of the liquidus and solidus temperatures of the alloys than data that would be obtained by cooling the melts. Theoretically, the melting range and freezing range of an alloy should be the same, but it is difficult to obtain an accurate cooling curve because the tendency for supercooling is generally greater than the tendency for superheating.

TABLE VI.—PATTERNMAKER'S SHRINKAGE.

Pouring Temp, deg Fahr	Gage Length, in.	Average Shrinkage, in. per ft[a]	Patternmaker's Shrinkage, in. per ft
		80-10-10 ALLOY	
1800 to 1865...	3	0.120	1/8
	6	0.132	1/8
	9	0.132	1/8
		Average 0.128	1/8 ± 1/64
		85-5-5-5 ALLOY	
1950....	3	0.168	11/64
	6	0.156	5/32
	9	0.168	11/64
		Average 0.164	11/64 ± 1/64
		NAVY "M" ALLOY	
1950 to 1960...	3	0.204	13/64
	6	0.180	3/16
	9	0.180	3/16
		Average 0.188	3/16 ± 1/64

[a] Standard deviation of the average values is ±0.024 in. per ft.

Patternmaker's Shrinkage:

A modification of a method described by Pilling and Kihlgren (4) was used for measuring patternmaker's shrinkage. Specimens were of the design illustrated by Fig. 3, which shows a 3-in. gage length. Actually, castings of three different gage lengths were used: 3, 6, and 9 in.

The shrinkage specimens were cast in dry sand molds composed of fine core sand. Just prior to casting, the gage length of each mold was measured to 0.001 in. with a micrometer caliper. The castings were then poured at as low a temperature as possible to minimize liquid shrinkage.

After the castings had cooled to room temperature, the gage lengths of the castings were measured to 0.001 in. with a micrometer caliper. The total patternmaker's shrinkage was equal to the difference between the gage length of the mold and the gage length of the casting. The results of these experiments, conducted on castings in duplicate, are presented in Table VI.

Apparently the length of the casting, within the range studied, had little effect on the patternmaker's shrinkage. The average difference in shrinkage between castings of the same alloy, but of different gage lengths, was less than 0.012 in. per ft. Thus, the estimated accuracy of these data is ±10 per cent.

Density and Specific Gravity:

Density measurements were made on machined tension-bar specimens before the bars were tested in tension. The density was determined from measurements obtained by weighing thoroughly cleaned specimens in air and then weighing them when suspended in chemically pure carbon tetrachloride. When measurements were made in carbon tetrachloride, the temperature of the bath was recorded so that a correction could be made to obtain the density of the bath at that temperature. From the data thus obtained, the density of the specimens was calculated from the following formula:

$$D_s = \frac{W_A \times D_L}{W_A - W_L}$$

where:

D_s = density of specimen, g per cu cm,

D_L = density of carbon tetrachloride, g per cu cm,

W_A = weight of specimen in air, g, and

W_L = weight of specimen in carbon tetrachloride, g.

Although the temperature of the bath and specimen ranged from 67 to 75 F, the correction for the density of the specimen at 68 F is of the order of 0.0001 g per cu cm. Since the density determinations are estimated to be accurate to within ±0.25 per cent (±0.02 g per cu cm), correction for the density at 68 F is insignificant. Therefore, the experimental data are reported as values obtained at 68 F, which is the accepted temperature for reporting such data.

TABLE VII.—DENSITY AND SPECIFIC GRAVITY.

Alloy	Density						Specific Gravity		
	g per cu cm			lb per cu in.					
	Maximum	Minimum	Average[a]	Maximum	Minimum	Average	Maximum	Minimum	Average
80-10-10	9.04	8.96	8.99	0.326	0.324	0.325	9.05	8.97	9.00
85-5-5-5	8.87	8.76	8.82	0.320	0.316	0.318	8.88	8.77	8.83
Navy "M"	8.71	8.62	8.64	0.315	0.311	0.312	8.72	8.63	8.65

[a] Average value for 5 specimens. Standard deviation for the average values is ±0.02 g per cu cm.

The density and specific gravity of the three alloys are presented in Table VII. The values for specific gravity were obtained by dividing the density value of the specimen by the density of water, which is 0.998405 g per cu cm at 68 F.

Electrical Resistivity:

The electrical resistivity of the three copper-base alloys was measured by the voltage-drop method described by ASTM Method B 193 – 57.[13] The specimens were cylinders $\frac{5}{16}$ in. in diameter and 7 in. long. The specimens were submerged in a thermostatically controlled oil bath that was stirred continually, and resistance measurements were made on both the heating and cooling portions of a thermal cycle. A constant temperature was maintained for approximately 15 min at each temperature at which resistance measurements were made. The voltage drop across 5 in. of the specimen's length was measured with pointed probes.

Values for the electrical resistivity of the three alloys at temperatures ranging from 68 to 450 F are given in Table VIII.

TABLE VIII.—ELECTRICAL RESISTIVITY AT VARIOUS TEMPERATURE LEVELS.[a]

Temperature, deg Fahr	Electrical Resistivity, microhm-cm		
	80-10-10	85-5-5-5	Navy "M"
68	17.01	11.43	12.04
150	17.63	11.87	12.46
200	18.00	12.13	12.71
250	18.38	12.42	12.97
300	18.76	12.70	13.22
350	19.15	12.99	13.48
400	19.53	13.28	13.74
450	19.92	13.59	13.99

[a] Only one specimen was tested for each alloy.

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[13] Method of Test for Resistivity of Electrical Conductor Materials (B 193 – 57), 1957 Supplement to Book of ASTM Standards, Part 2, p. 17.

These values are estimated to be accurate to within ±0.5 per cent. The data show that resistivity increases a slight amount as the temperature is increased. The electrical conductivities, expressed as a per cent of the International Annealed Copper Standard, are presented in Table IX.

Thermal Conductivity:

The thermal conductivity of the alloys was measured in an apparatus that was essentially the same as the one described by Van Dusen and Shelton (5). Briefly, the method consists of heating one end of a specimen, measuring the temperature gradients along the specimen, and determining the rate of heat flow through it and into an ingot iron standard soldered to the opposite end of the test specimen. The end of the test specimen was heated while the end of the ingot iron standard was cooled. Radial heat flow into or away from the specimens was minimized by thermal insulation and an encircling guard tube in which the temperatures were adjusted, as nearly as possible, to match those in the specimen and the standard at corresponding positions.

TABLE IX.—ELECTRICAL CONDUCTIVITY AT VARIOUS TEMPERATURE LEVELS.[a]

Temperature, deg Fahr	Electrical Conductivity, per cent IACS[b]		
	80-10-10	85-5-5-5	Navy "M"
68..........	10.14	15.08	14.32
150..........	9.78	14.52	13.84
200..........	9.58	14.21	13.56
250..........	9.38	13.88	13.29
300..........	9.19	13.58	13.04
350..........	9.00	13.27	12.79
400..........	8.82	12.98	12.55
450..........	8.65	12.68	12.32

[a] Only one specimen was tested for each alloy.

[b] The resistivity of the International Copper Standard is 1.7241 microhm-cm.

The test specimens were cylinders 0.787 in. in diameter and 5.906 in. in length. The ingot iron standard was 1.969 in. long. Four thermal-conductivity values were calculated, each at a different mean temperature for each thermal equilibrium. Data were recorded for three or four equilibria in each specimen.

The values obtained for thermal conductivity are given in Table X. Because of the uncertainty of the thermal conductivity of the ingot iron used as the reference material, it is estimated that the data are accurate to within ±5 per cent.

TABLE X.—THERMAL CONDUCTIVITY AT VARIOUS TEMPERATURES.[a]

Temperature, deg Fahr	80-10-10		85-5-5-5		Navy "M"	
	Btu ft per hr sq ft deg Fahr	Per Cent of Copper	Btu ft per hr sq ft deg Fahr	Per cent of Copper	Btu ft per hr sq ft deg Fahr	Per Cent of Copper
68...	27.5	12.1	41.6	18.3	40.2	17.7
100...	27.7	12.2	42.4	18.6	40.7	17.9
150...	28.8	12.6	43.8	19.2	41.7	18.3
200...	29.7	13.0	45.2	19.8	42.8	18.8
250...	30.6	13.4	46.8	20.6	44.0	19.3
300...	31.7	13.9	48.5	21.3	45.5	20.0
350...	32.8	14.4	50.3	22.1	47.3	20.8
400...	34.0	14.9	52.5	23.1	49.4	21.7
450...	35.4	15.6	54.6	24.0	50.4	22.1

[a] Only one specimen was tested for each alloy.

TABLE XI.—MEAN LINEAR THERMAL EXPANSION COEFFICIENTS FOR VARIOUS TEMPERATURE RANGES.[a]

Temperature Range, deg Fahr	Coefficient, 10^{-6} in. per in. per deg Fahr		
	80-10-10	58-5-5-5	Navy "M"
68 to 150.....	9.88	9.75	9.27
68 to 200.....	10.00	9.85	9.43
68 to 250.....	10.11	9.95	9.60
68 to 300.....	10.17	10.00	9.74
68 to 350.....	10.25	10.04	9.84
68 to 400.....	10.30	10.06	9.95
68 to 450.....	10.35	10.16	10.08
68 to 500.....	...	...	10.15
68 to 550.....	...	...	10.25

[a] Only one specimen was tested for each alloy.

Thermal Expansion:

Linear thermal expansion of the three alloys was measured on cylindrical specimens, $\frac{5}{16}$ in. in diameter and 3 in. long. The specimen was placed in the bottom of a quartz tube positioned in a vertical, electrically heated furnace. A quartz rod rested on the specimen and extended through the top of the furnace, where it activated a dial indicator. Before each determination was started, the quartz tube containing the specimen was evacuated to a pressure of about 3×10^{-5} mm of mercury to prevent oxidation, as well as to minimize heat loss by convection. The maximum heating and cooling rates were approximately 5 F per min. The sensitivity of the dial gage was 0.0001 in.

Table XI gives the mean linear thermal expansion coefficients of each alloy over the temperature ranges of 68 to 150 F and 68 to 550 F. These coefficients are estimated to be accurate to within ±2 per cent.

Acknowledgment:

The authors are grateful to the Brass and Bronze Ingot Inst., the sponsor of this program, for permission to publish the data. Appreciation is expressed to the various members of the Brass and Bronze Ingot Inst. for supplying the ingot metal and for conducting the analytical work for chemical composition of the castings. Battelle Memorial Institute staff members who conducted specialized tasks were R. M. Lang:—melting, casting, measurement of patternmaker's shrinkage, impact properties, hardness measurements, and density measurements; J. E. Campbell:—tensile and compression properties; W. S. Hyler and E. D. Abraham:—fatigue properties; H. L. Shaw:—machinability determinations; A. A. Bauer:—melting-range determinations; H. W. Deem:—measurements of electrical resistivity, thermal conductivity, and thermal-expansion properties; F. J. Barone:—statistical treatment of the data; and C. H. Lorig:—consultation and guidance.

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[See Joint Discussion, p. 805.—ED.]

THE CREEP PROPERTIES OF THREE LOW-SHRINKAGE, COPPER-BASE CASTING ALLOYS*

By Ward F. Simmons[1] and J. G. Kura[1]

Synopsis

A creep evaluation was conducted on cast specimens of 80-10-10, 85-5-5-5, and Navy "M" alloys at 350, 450, and 550 F. Most of the tests were conducted for a period of 2000 hr. At one temperature for each alloy, the creep tests were carried out to 10,000 hr duration. Creep-rupture tests were also made at this temperature.

At 350 F, the highest creep resistance was exhibited by Navy "M" alloy. At 450 F, the Navy "M" and 85-5-5-5 alloys were about equal. Navy "M" and 85-5-5-5 alloys were also about equal in creep resistance at 550 F, but the Navy "M" alloy appeared to be more stable than the 85-5-5-5 alloy at this temperature.

Short-time, room-temperature tension tests were made on creep-tested specimens to determine the residual tensile properties after the long-time exposure to stress and temperature. The results of these tests indicated that creep exposure produced a moderate increase in yield strength for all three alloys, regardless of the specimen's previous history. The ultimate tensile strength and ductility, however, showed significant deleterious effects from prior exposure under certain conditions.

This paper presents the creep and creep-rupture data for the same three cast copper-base alloys discussed in a companion paper.[2] The creep evaluation was conducted at Battelle Memorial Institute as a part of a continuing research program sponsored by the Brass and Bronze Ingot Inst. (BBII). The work was initiated by the BBII to provide accurate determinations of various physical and mechanical properties, including creep, of sound test bars from good-quality melts of the 80-10-10, 85-5-5-5, and Navy "M" alloys. In the past very few creep and creep-rupture data have been available for these casting alloys (**1, 2**).[3] The authors are indebted to the BBII for permission to publish the results of this work.

Details of the foundry practice used in preparing and establishing the quality of the melts, as well as the various physical and mechanical properties, other than creep, are given in the companion paper. The specimens for the creep portion of the program were machined from Double Horizontal Full-Web type test-bar castings. This test-bar casting (Fig. 2 of ASTM Recommended Practice: B 208 – 54),[4] is recommended by the ASTM for low-shrinkage alloys.

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* Presented at the Sixty-first Annual Meeting of the Society, June 22–27, 1958.

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[2] J. G. Kura and R. M. Lang, "Mechanical and Physical Properties of Three Low-Shrinkage, Copper-Base Casting Alloys," p. 775 this publication.

[3] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 804.

Table I gives the range of chemical composition for the castings tested. The ASTM has designated the following specifications for these alloys: 80-10-10, ASTM Specification B 144 – 52,[5] alloy 3(A); 85-5-5-5, ASTM Specification B 62 – 52;[6] Navy "M," ASTM Specification B 143 – 52,[7] alloy 2(A). The chemical analyses of all castings were well within the ASTM specifications.

TABLE I.—CHEMICAL COMPOSITIONS FOR 80-10-10, 85-5-5-5, AND NAVY "M" ALLOYS.

Alloy	Composition, weight per cent							
	Copper	Tin	Lead	Zinc	Iron	Antimony	Nickel	Phosphorus
80-10-10[a]	79.00 to 79.75	9.19 to 9.54	9.86 to 10.38	0.26 to 0.29	0.01 to 0.02	0.23 to 0.26	0.42 to 0.46	0.005 to 0.01
85-5-5-5[a]	84.78 to 85.13	4.32 to 4.48	4.86 to 5.33	4.11 to 4.85	0.10 to 0.12	0.16 to 0.17	0.66 to 0.68	0.006 to 0.014
Navy "M"[a]	87.19 to 88.42	5.5 to 5.90	1.30 to 1.46	3.82 to 4.70	0.06 to 0.10	0.13 to 0.15	0.53 to 0.56	0.009 to 0.013

[a] The range given is the spread in analysis of all castings tested.

The program on creep and creep-rupture testing was carried out within the temperature range of 350 to 550 F. The test temperatures used were selected on the basis of the creep strengths exhibited by the various alloys as the evaluation program developed. An attempt was made to select the highest temperature at which useful data for service application might be obtained for each alloy. The specific test temperatures used were as follows:

Alloy	Temperature, deg Fahr
80-10-10	350, 450[a], 550
85-5-5-5	350, 450[a], 550
Navy "M"	350, 450, 550[a]

[a] Creep-rupture tests and a 10,000-hr test were made at this temperature.

The stress to produce a creep rate of 0.00001 per cent per hr, along with four fifths of the stress to produce rupture in 100,000 hr, is used by the ASME Boiler and Pressure Vessel Code Committee as a basis for establishing maximum allowable stresses for nonferrous materials in the temperature range where significant creep occurs. Previous experience has indicated that the creep strengths for a creep rate of 0.00001 per cent per hr are usually lower than the 100,000-hr rupture strengths and are much more easily determined. Therefore, the principal criterion in the present investigation was creep strength, and tests were made to determine the stress to produce a creep rate of 0.00001 per cent per hr for each alloy at the temperatures indicated above.

Creep-rupture tests were made to determine the stress to produce rupture in 1000 hr at one temperature for each alloy. A few rupture tests were also made at other temperatures. One long-time creep test of 10,000 hr duration was made for each alloy at the same temperature as the rupture tests.

Second-stage creep rates can be determined just as well from 2000-hr creep tests as from 10,000-hr tests, assuming, of course, that second-stage creep is reached in less than 2000 hr. The purpose of the 10,000-hr creep tests was (*a*) to give an indication of the long-time, high-temperature stability of the alloys, (*b*) to obtain long-time data for 0.1 per cent plastic strain, and (*c*) to obtain long-time data for the time of transition to third-stage creep. For this purpose, it was believed that long-time creep tests would yield more useful data than would 10,000-hr rupture tests for the same expenditure of time. This belief was confirmed by the data obtained.

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[4] Recommended Practice for Tension Test Specimens for Copper-Base Alloys for Sand Castings (B 208 – 54), 1955 Book of ASTM Standards, Part 2, p. 345.

[5] Specification for High-Leaded Tin-Bronze Sand Castings (B 144 – 52), 1955 Book of ASTM Standards, Part 2, p. 233.

[6] Specification for Composition Brass or Ounce Metal Castings (B 62 – 52), 1955 Book of ASTM Standards, Part 2, p. 119.

[7] Specification for Tin-Bronze and Leaded Tin-Bronze Sand Castings (B 143 – 52), 1955 Book of ASTM Standards, Part 2, p. 229.

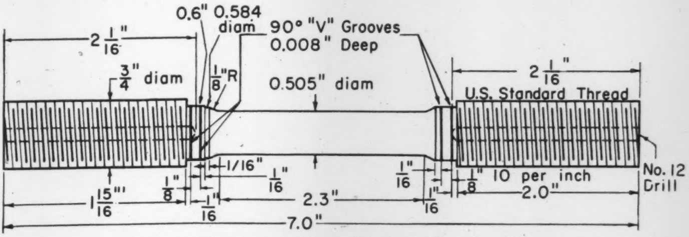

FIG. 1.—Creep and Creep-Rupture Test Specimen.

Both creep data and creep-rupture data are useful in the design of equipment intended for long-time service at elevated temperatures. Creep data are most useful in the design of long-time equipment, such as steam-generation equipment, where most of the deformation is expected to take place by creep during a service lifetime of 20 yr or more.

Stress-rupture tests were employed extensively in the early nineteen forties to evaluate high-temperature alloys and to supply design data for short-time applications, such as for jet engine parts. At the present time, data from the more refined creep-rupture tests are being used for evaluation and design for relatively short lifetimes. In addition to requiring data on the stresses to produce rupture in 100 or 1000 hr, designers are now requesting data on the stresses to produce some small amount of plastic strain in a given time, for example, 0.1 or 0.2 per cent plastic strain in 1000 hr. Of course, long-time creep data are still needed in the design of equipment intended for long-time service at high temperatures. Both the short-time and the long-time creep data can be conveniently presented as isochronous stress-strain curves, as has been done in this paper.

EXPERIMENTAL WORK

Test Equipment and Method of Test:

The creep and creep-rupture tests were made in standard Battelle creep-testing machines. These machines load the specimen by means of a 9 to 1 lever arm. The specimen is contained in a 6-in.-diameter electric furnace wound with 14-gage Chromel-A wire and insulated with Sil-O-Cel.

The desired test temperature is maintained in the creep furnace to within ±2 F by means of either a Tag Celectray Controller equipped with a throttling mechanism for closer control, or a Foxboro Controller to which an anticipating device has been added to improve the temperature control. The test temperatures are checked daily with a semiprecision potentiometer and adjusted, if necessary. The creep and creep-rupture tests were conducted in accordance with ASTM Recommended Practice E 22 – 41,[8] except that closer tempera-

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[8] Recommended Practice for Conducting Long-Time High-Temperature Tension Tests of Metallic Materials (E 22 – 41), 1955 Book of ASTM Standards, Part 1, p. 1612.

TABLE II.—DETAILED CREEP AND CREEP-RUPTURE DATA.

Temperature, deg Fahr	Stress, ksi	Duration, hr	Initial Strain, per cent	Total Strain, per cent	Contraction Upon Release of Load, per cent	Creep Rate		Transition to Third-Stage Creep		Elongation, per cent	Reduction Area, per cent
						Second Stage, per cent hr	Final, per cent per hr	Time, hr	Plastic Strain, per cent		
						80-10-10 Alloy					
350....	30.0	17.5[a]	15.0	...	...	...	...	...	...	18.2	23.1
350....	20.0	2 022	3.30	5.17	0.270	0.00036	0.00036	>2022	...	...	...
350....	15.0	2 016	0.550	0.878	0.158	0.000070	0.000070	>2016	...	...	...
350....	12.0	1 827	0.163	0.235	0.136	0.000020	0.000020	>1827	...	...	...
450....	20.0	50[a]	3.4	...	...	0.065	...	...	...	7.6	9.1
450....	17.0	270[a]	2.0	...	...	0.0043	...	230	3.8	5.5	5.8
450....	16.0	564[a]	0.95	...	...	0.0020	...	450	2.4	3.5	5.6
450....	12.0	1 514	0.182	0.575	0.151	0.000185	0.000185	>1514	...	...	...
450....	10.0[b]	2 015	0.100	0.428	0.106	0.000045	...	>2015	...	...	...
450....	8.0[b]	10 024	0.099	0.358	0.089	0.000015	0.000030	6700	0.16	...	...
550....	8.0	625	0.098	0.559	0.049	0.00047	0.00115	200	0.17	...	...
550....	6.5	1 224	0.076	0.718	0.072	0.00032	0.00074	325	0.12	...	...
550....	5.0	2 020	0.047	0.544	0.052	0.00019	0.00026	1100	0.25	...	...
550....	3.0[b]	2 021	0.037	0.137	0.033	0.000040	0.000040	>2021	...	...	...
						85-5-5-5 Alloy					
350....	28.0	32[a]	20.4	...	...	0.02	...	...	...	21.9	22.2
350....	24.0	717	13.8	13.89	0.242	0.00010	0.00010	>717	...	...	...
350....	20.0	1 015	7.86	9.45[d]	0.212	0.00007	0.00007	>1 015	...	...	...
350....	12.0	1 533	0.446	0.477	0.121	0.000010	0.000010	>1 533	...	...	...
350....	11.0	1 800	0.225	0.250	0.111	0.000003	0.00003	>1 800	...	...	...
450....	20.0	52[a]	7.95	...	...	0.0145	...	...	...	11.5	16.6
450....	17.5	259[a]	5.02	...	...	0.0022	...	162	5.5	8.2	13.8
450....	15.5	1 083[a]	2.75	...	...	0.00063	...	...	...	6.7	13.5
450....	15.5	532[c]	1.84	...	0.152	...	...	...	...	...	...
450....	12.0[b]	2 022	0.495	0.650	0.121	0.000020	0.000020	>2 022	...	...	...
450....	10.0[b]	10 000	0.142	0.230	0.108	0.000003	0.000003	>10 000	...	...	...
550....	11.0	412[a]	0.265	...	...	0.0002	...	280	0.27	3.1	6.7
550....	10.0	744[a]	0.158	...	...	0.000105	...	625	0.16	1.8	6.5
550....	8.0[b]	2 018	0.102	0.168	0.090	0.000024	0.000024	>2 018	...	...	...
						Navy "M" Alloy					
350....	30.0	717	18.85	19.0	0.392	0.00015	0.00025	350	18.50	...	...
350....	20.0	917	6.59	8.82[e]	0.210	0.00004	0.00004	>917	...	...	...
350....	15.0	1 847	1.86	1.90	0.138	0.000007	0.000007	>1847	...	...	...
350....	12.0	1 533	0.204	0.218	0.108	0.000001	0.000001	>1533	...	...	...
450....	15.0	94[f]	2.538	2.67	0.165	...	...	...	...	...	...
450....	13.0	2 018	1.134	1.335	0.138	0.000038	0.000038	>2018	...	...	...
450....	12.5	1 200	0.256	0.316	0.116	0.000025	0.000025	>1200	...	...	...
450....	12.0[b]	2 009	0.360	0.433	0.125	0.000013	0.000013	>2009	...	...	...
550....	20.0	2.8[a]	6.48	...	...	...	...	...	...	9.0	13.1
550....	15.0	58[a]	1.65	...	...	0.087	...	...	...	5.4	14.4
550....	12.0	852[a]	0.311	...	...	0.00024	...	650	0.41	3.6	6.1
550....	10.0[b]	2 181	0.105	0.374	0.098	0.000075	0.00016	1100	0.10	...	...
550....	8.0	1 176	0.076	0.121	0.068	0.000030	0.000030	>1176	...	...	...
550....	7.0[b]	10 000	0.067	0.405	0.073	0.000017	0.000047	2500	0.06	...	...

[a] Rupture time.

[b] A room-temperature tensile test was made on this specimen after creep testing.

[c] Discontinued because of high strain resulting from failure in an adjacent creep machine.

[d] Total strain includes 1.41 per cent which resulted from the rupture of a test specimen in an adjacent creep machine.

[e] Total strain includes 2.04 per cent which resulted from the rupture of a test specimen in an adjacent creep machine.

[f] Discontinued because plastic strain on loading was higher than desired.

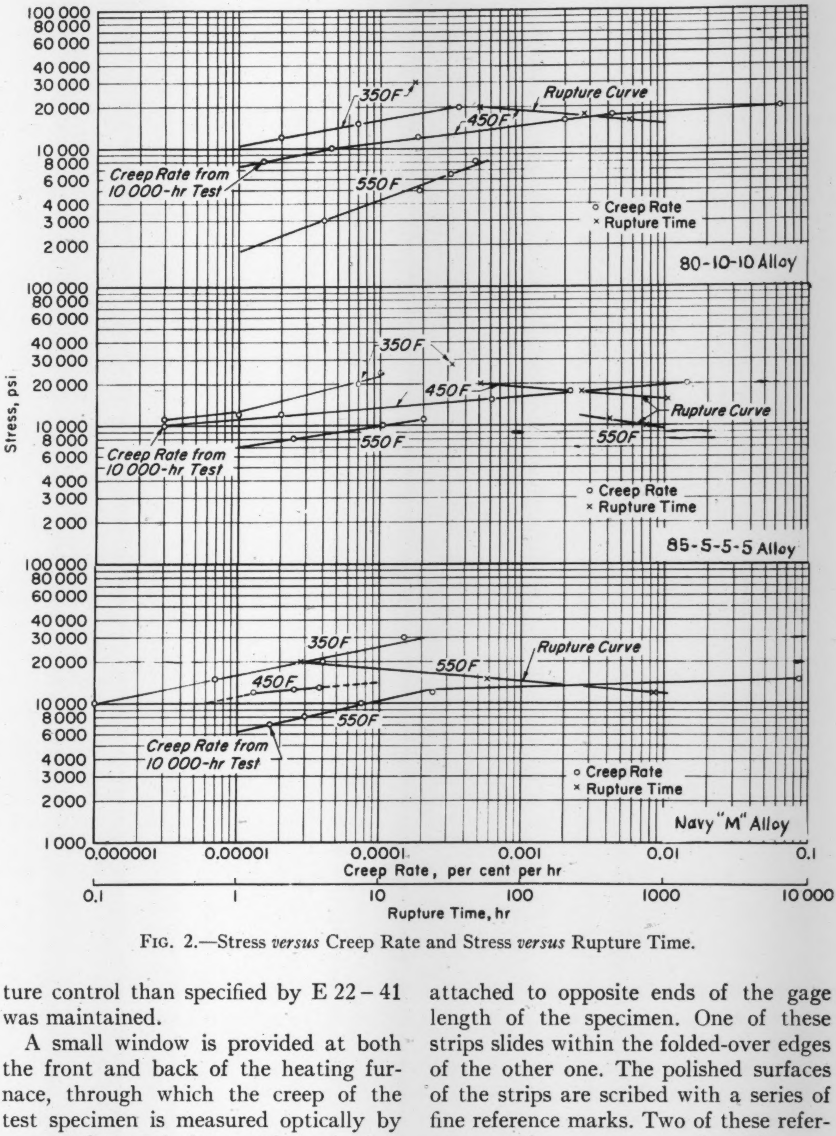

FIG. 2.—Stress *versus* Creep Rate and Stress *versus* Rupture Time.

ture control than specified by E 22 – 41 was maintained.

A small window is provided at both the front and back of the heating furnace, through which the creep of the test specimen is measured optically by means of two platinum-strip extensometers and a filar microscope. Each extensometer consists of two 0.005-in. thick strips of platinum-rhodium foil that are attached to opposite ends of the gage length of the specimen. One of these strips slides within the folded-over edges of the other one. The polished surfaces of the strips are scribed with a series of fine reference marks. Two of these reference marks, one on each strip, are chosen, and the distance between them is measured with the filar microscope periodically during the test. The creep of the specimen is determined by measuring the change in the distance between the reference marks on the two platinum extensometer strips. Two observers read each test, and the average value of their strain measurements from the two extensometers is used in plotting the creep of the test specimens.

TABLE III.—SUMMARY OF CREEP AND RUPTURE STRENGTHS FOR 80-10-10, 85-5-5-5, AND NAVY "M" ALLOYS.

Alloy	Temperature, deg Fahr	Creep Strength, ksi		Rupture Strength, ksi	
		Creep Rate of 0.00001 per cent per hr	Creep Rate of 0.0001 per cent per hr	100 hr	1000 hr
80-10-10.....	350	10.4	15.9	...	...
	450	7.4	11.0	18.8	15.0
	550	1.8	4.2	...	...
85-5-5-5.....	350	12.5	22.3	...	...
	450	11.1	13.5	19.0	15.4
	550	7.0	9.9	...	9.6
Navy "M"...	350	16.0	25.6	...	...
	450	11.2	14.1	...	...
	550	6.2	10.2	14.2	11.7

The filar microscope has a magnification power of 20× and is mounted on a vertical screw. The smallest division on its filar eyepiece is 0.00005 in., which, with a specimen of 2.3-in. gage length, is equal to 0.0022 per cent.

The specimen used for both the creep and the creep-rupture tests is shown in Fig. 1. It is 7 in. long and 0.505 in. in diameter at the reduced section and has a 2.3-in. gage length.

When making a test, the specimen and extensometer are assembled and placed in the creep machine. The assembly is heated in the furnace to the desired test temperature and held about 1 hr before load is applied to the specimen. The zero reading upon which all strain measurements are based is made at the test temperature with no load on the specimen.

TEST RESULTS

The detailed creep and creep-rupture data are given in Table II and the creep rates and rupture times are plotted in Fig. 2. A summary of the creep strengths for creep rates of 0.00001 and 0.0001 per cent per hr is given in Table III. The rupture strengths for rupture in 100 or 1000 hr are also summarized in Table III. An examination of these data reveals several interesting facts about the various alloys. At 350 F, Navy "M" alloy exhibited higher creep strengths than did the other two alloys, but at 450 F, the load-carrying abilities of alloys 85-5-5-5, and Navy "M", as revealed by their creep resistance, appear to be about equal. This is also true at 550 F However, at 550 F the 85-5-5-5 alloy showed a marked tendency to fail with a brittle fracture in relatively short times. The 85-5-5-5 alloy specimen tested at 550 F and 10,000 psi (Table II), which had a minimum creep rate of 0.000105 per cent per hr, ruptured in 744 hr with 1.8 per cent elongation. The test temperature of 550 F is only about 70 F below the melting point of the lead phase in the 85-5-5-5 alloy. Therefore, it is not surprising to observe that brittle failure occurs in relatively short times at 550 F. Navy "M" appears to be the best alloy for service at 550 F, but this is a very high temperature for any of these alloys.

10,000-Hr Creep Tests:

The 10,000-hr creep tests were made to obtain information on the high-temperature stability of the alloys. A summary of the significant data is given below:

Alloy	Temperature, deg Fahr	Stress, psi	Creep Rate, per cent per hr		Transition to Third-Stage Creep	
			Second Stage	Final	Hr	Plastic Strain, per cent
80-10-10..........	450	8 000	0.000015	0.000030	6 700	0.16
85-5-5-5..........	450	10 000	0.000003	0.000003	>10 000	>0.12
Navy "M".......	550	7 000	0.000017	0.000047	2 500	0.06

These long-time tests demonstrated that the increase in the creep rate at the end of second-stage creep took place very slowly, and the transition point could not be determined by inspection. The transition points were selected arbitrarily by drawing a line on the time-deformation graph parallel to the minimum creep rate and 0.01 per cent above it. The intersection of this line with the creep curves was defined as the transition point.

Data for 0.1 and 0.5 per cent plastic strain, the transition points, and rupture times have been plotted in Fig. 3. These "design curves," which extend out to fairly long times, give a relatively complete picture of the strength properties of the alloys at the temperatures for which the data are most complete. The shapes of the curves for the 85-5-5-5 alloy at 450 F, in Fig. 3, are most nearly ideal, with the transition-point curve parallel and fairly close to the rupture curve. A material must always be used with caution under conditions of time or stress above the transition-point curve. Notice that the strain at the transition point decreases with an increase in time, as does the rupture strain. Thus, if failure occurs after very long times, it will occur at a low value of strain.

The valuable low-stress data for 0.1 per cent plastic strain and the transition points in Fig. 3 were obtained by selecting 2000-hr creep tests and continuing them for 10,000 hr. If 10,000-hr rupture tests had been made instead of the 10,000-hr creep tests, a rupture point at about 10,000 hr and a transition point at a much shorter time would have been obtained. Such data would not have been so useful for design purposes as the data that were actually obtained. Also, unbroken specimens exposed under stress for 10,000 hr would not have been available for the tests described in the last section of this paper.

Comparison of Short-Time Yield Strength and Creep Strengths:

The effect temperature has on (*a*) the yield strength at 0.5 per cent extension under load, (*b*) the creep strengths for 0.00001 and 0.0001 per cent per hr creep rates, and (*c*) the strength for 0.5 per cent plastic strain in 1000 hr is shown for each of the three alloys in Fig. 4. These curves are helpful in determining the temperature at which creep becomes significant. They are particularly helpful when short-time properties may no longer be adequate for design purposes. Notice that at the lower temperatures the creep curves are above the yield strength curve, but at high temperatures the creep curves cross the yield strength curve and fall below it. The temperature at which the curve for a creep rate of 0.00001 per cent per hr crosses the yield strength curve and lies beneath it may

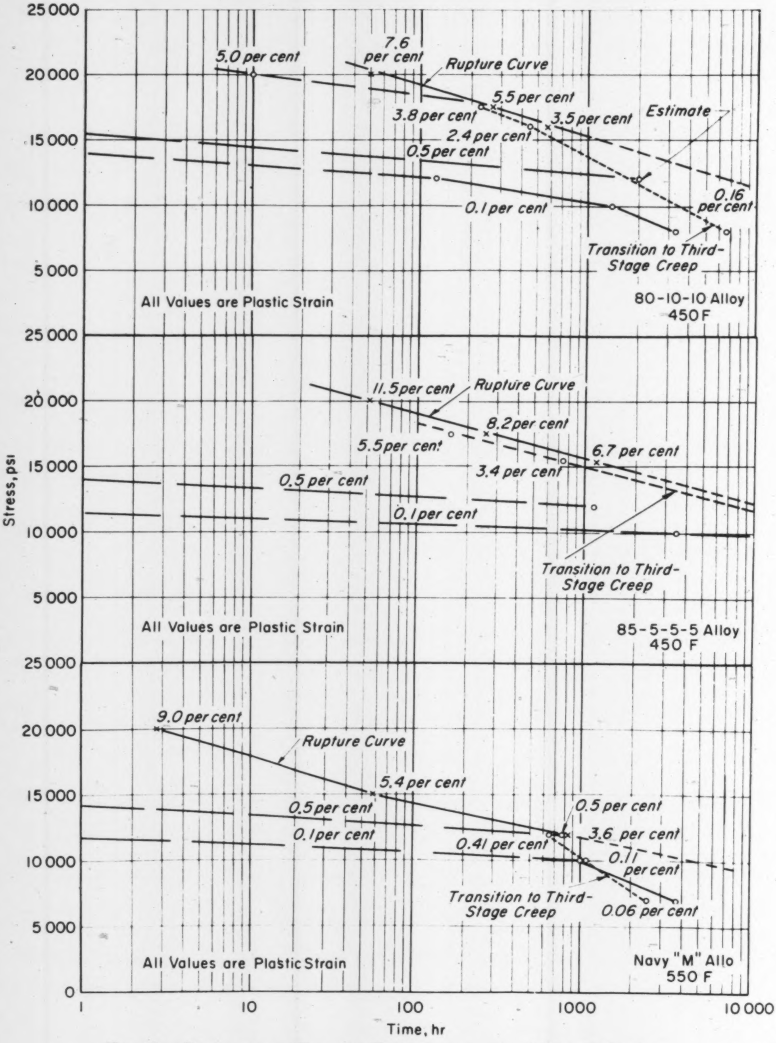

FIG. 3.—Stress *versus* Time for Various Amounts of Plastic Strain.

be considered as the temperature at which creep begins to become significant. However, if the design stress is based on some fraction of the yield strength, for example, two thirds of it, the temperature above which the creep curve would control the design would be at the intersection of a curve representing two thirds of the yield strength and the curve for a creep rate of 0.00001 per cent per hr. Thus, when designing on the basis of two thirds of the yield strength, it may be possible to use short-time tensile data for a limited temperature range above that temperature at which creep becomes significant. However, even where this is possible, the creep data are needed to define the maximum temperature at which the short-time properties can be used.

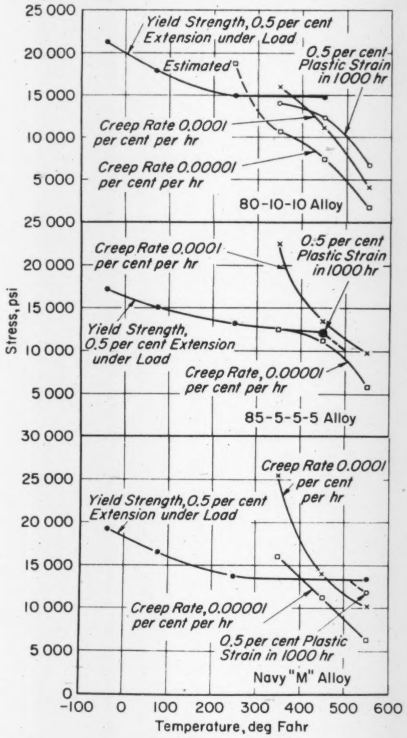

FIG. 4.—Stress *versus* Temperature for Yield and Creep Strengths.

It may be helpful to outline briefly the basis used by the ASME Boiler and Pressure Vessel Code for establishing maximum allowable stress values for nonferrous materials. The following excerpt is from Appendix Q of Section VIII of the above code, "Rules for Construction of Unfired Pressure Vessels" (1956):

"Except for bolting materials, the maximum allowable stress values may be determined as the lowest of the following when the tensile and yield strengths are obtained from standard short-time tests:

"1. One fourth of the tensile strength as adjusted to minimum.

"2. Two thirds of the yield strength (as defined in the material specification) as adjusted to minimum.

"3. The stress producing a secondary creep rate of 0.1 CRU (creep rupture unit) or 0.1 per cent in 10,000 hr (0.00001 per cent per hr).

"4. Four fifths of the stress producing rupture in 100,000 hr.

"The tensile and yield strengths are adjusted to minimum by multiplying the test results by the ratio:

$$\text{``}\frac{\text{(Minimum specified or expected tensile or yield strength)}}{\text{(Actual room-temperature tensile or yield strength for the lot of material under test)}}\text{''}$$

For an annealed material, the design stress at room temperature and at moderately elevated temperatures is usually based on the yield strength. For a material that has improved mechanical properties due to cold work, the yield strength is increased to a greater degree than the tensile strength, and the design stress may then be based upon the tensile strength. However, castings normally are not cold worked prior to use. At temperatures within the creep range, where creep is significant or is the controlling factor, the design stress may be governed by either the rupture strength or the creep strength. As a rule, however, the lower stress values will be given by the creep-strength data.

Isochronous Stress-Strain Curves:

When sufficient data are available, the creep properties of a material can be illustrated by the use of isochronous stress-strain curves. These curves, shown

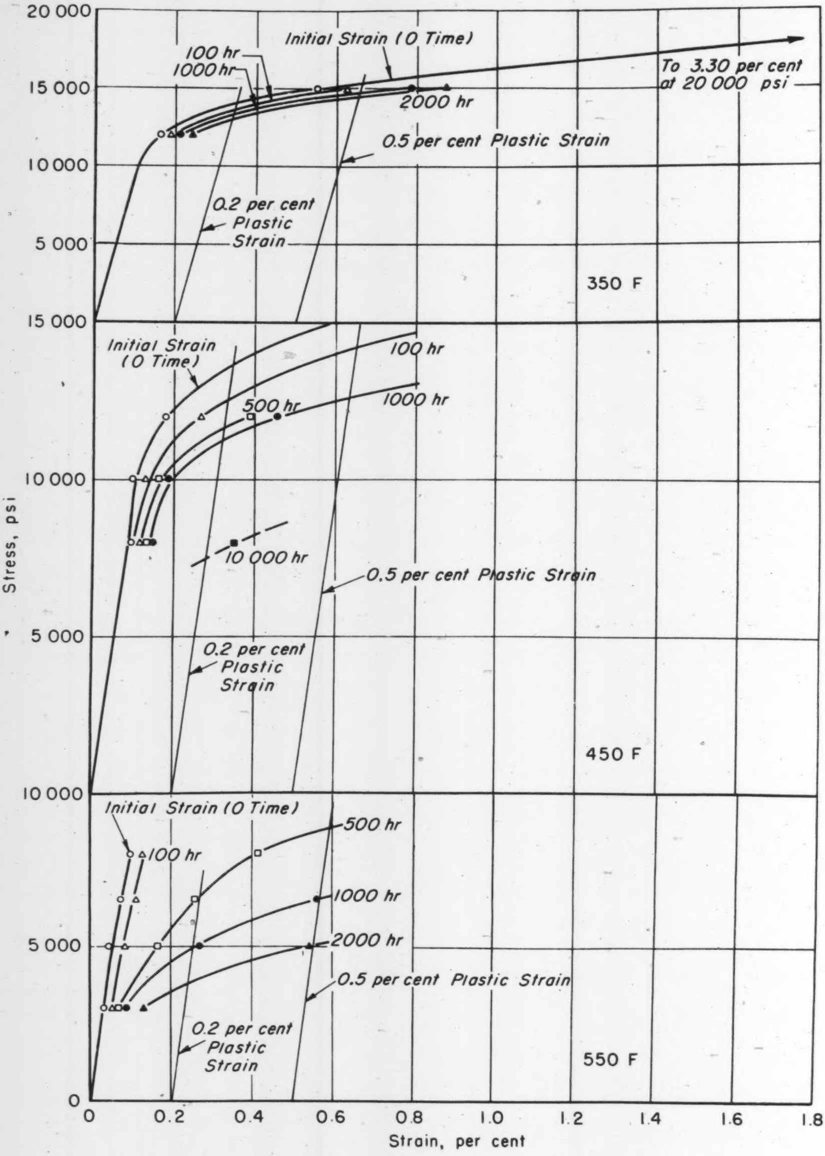

FIG. 5.—Isochronous Stress-Strain Curves for 80-10-10 Alloy.

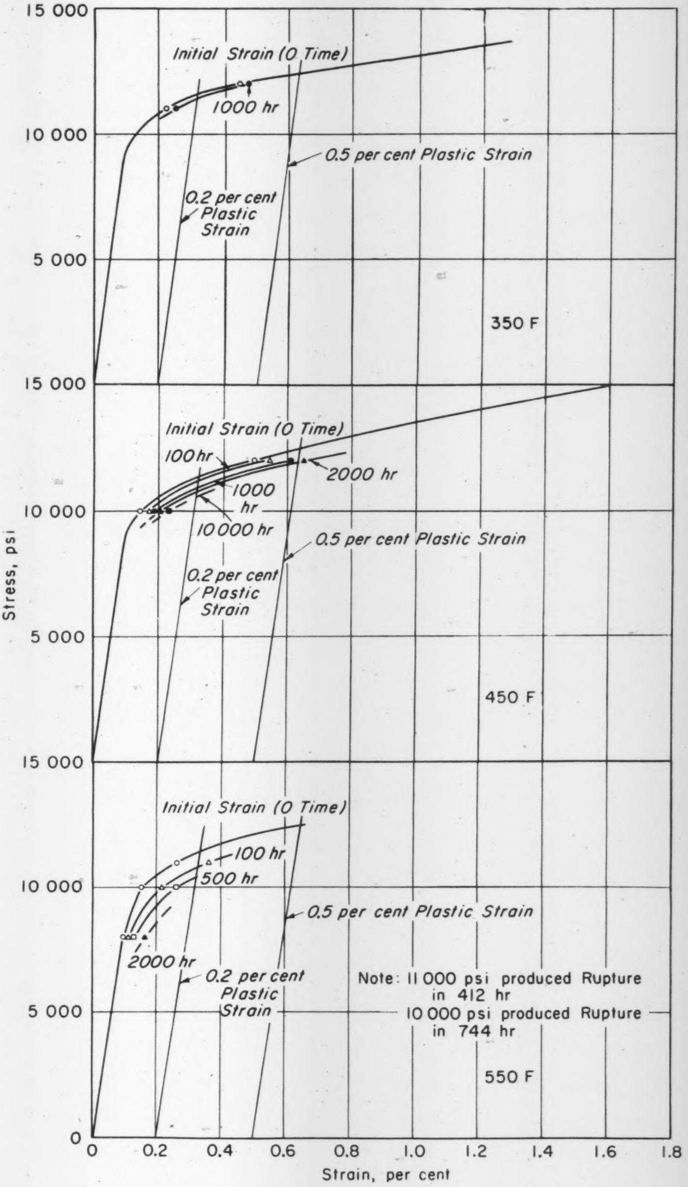

FIG. 6.—Isochronous Stress-Strain Curves for 85-5-5-5 Alloy.

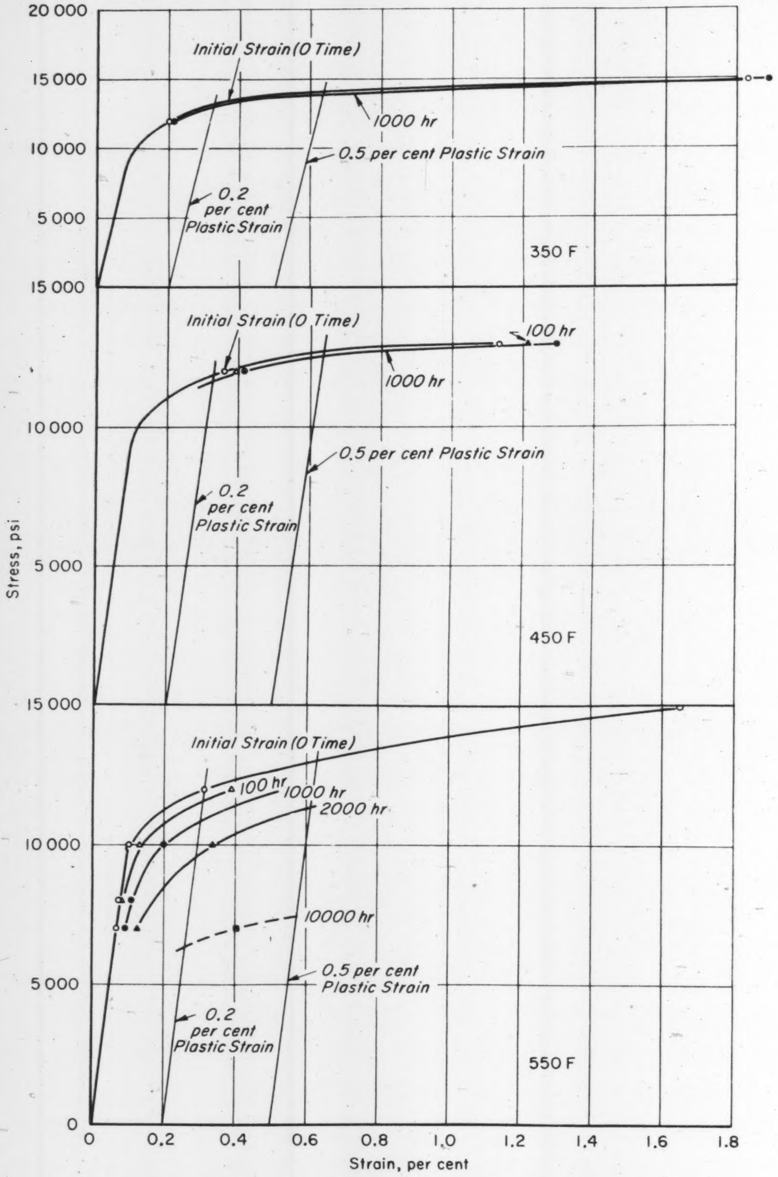

FIG. 7.—Isochronous Stress-Strain Curves for Navy "M" Alloy.

in Figs. 5, 6, and 7 for the three alloys, have the familiar shape of conventional stress-strain curves but include the effect of time.

The curves are prepared by plotting total-deformation data from the creep curves for various time periods of interest. These isochronous curves represent the stress-strain relationship of a material at a constant temperature and for a given time period. The upper curve in Figs. 5, 6, and 7, marked "initial strain (0 time)," was obtained from the initial strain that occurred upon the loading of the creep test specimens. This curve should have the same shape, and should yield essentially the same data, as the stress-strain curve from a conventional short-time tension test.

From these curves, it is a simple matter to determine the total deformation or strain to be expected at some given stress and time. Or, if the stress to produce a certain plastic strain in a given time is desired, for example, 0.2 per cent plastic strain in 2000 hr, a line drawn parallel to the modulus line through 0.2 per cent strain will yield the desired value at the intersection with the 2000-hr curve. In Fig. 5, this value is 13,000 psi for 80-10-10 alloy at 350 F. From the same figure, the stress to produce 0.5 per cent plastic strain in 2000 hr is 14,500 psi.

TABLE IV.—ROOM-TEMPERATURE, TENSION-TEST DATA ON UNBROKEN CREEP SPECIMENS.

Creep-Test Conditions				Yield Strength, ksi		Ultimate Strength, ksi	Elongation, per cent in 2 in.	Reduction in Area, per cent	Young's Modulus, 10^6 psi	Fracture
Alloy	Temperature, deg Fahr	Stress, ksi	Duration, hr	0.2 per cent Offset	0.5 per cent Extension[c]					
80-10-10.....	BEFORE CREEP TESTING[a]			17.3	17.9	39.0	30	24	11.6	...
	450	10.0	2 015	21.7	22.4	37.3	11.6	13.2	12.5	Uniformly gray
	450	8.0	10 024	21.4	22.4	27.2	1.8	4.1	10.7	Small amount of coppery area
	550	3.0	2 021	21.4	22.6	36.6	10.0	13.0	12.1	Uniformly gray
85-5-5-5	BEFORE CREEP TESTING[a]			14.4	15.1	35.2	32	30	12.4	...
	450	12.0	2 022	17.0	17.5	33.8	24.3	21.2	13.2	Small amount of coppery area
	450	10.0	10 000	16.7	17.5	34.1	26.2	23.8	13.2	Small amount of coppery area
	550	8.0	2 018	16.0	16.8	19.0	4.3	9.4	10.0	Small amount of coppery area[b]
Navy "M"...	BEFORE CREEP TESTING[a]			15.7	16.4	40.6	46	40	13.1	...
	450	12.0	2 009	17.2	17.7	37.9	41.8	31.6	11.7	Uniformly coppery, granular
	550	10.0	2 181	20.2	20.9	24.4	5.8	12.1	13.4	Uniformly coppery, some columnar grains
	550	7.0	10 000	21.0	21.8	23.2	3.0	8.0	13.5	Uniformly coppery, columnar

[a] Average values from 18 tests.

[b] Oxidized crack evident in the fracture. Low room-temperature tensile properties are the result of this crack, which was initiated during creep testing.

[c] Under load.

Room-Temperature Tensile Properties of Specimens Subjected to Creep Testing:

To determine the effect that long-term exposure to stress and temperature has on room-temperature properties, certain selected unbroken, creep-tested specimens were subsequently tested in short-time tension at room temperature. The specimens selected included the 10,000-hr test specimens for each alloy and a 2000-hr test specimen at the same temperature. Another 2000-hr creep specimen representing a second temperature was also tested. Thus, for each alloy, three specimens representing the two higher test temperatures were used to determine the residual tensile properties. The results of these tests are given in Table IV.

Prior exposure to elevated temperatures during creep testing produced a moderate increase in yield strength for each specimen tested, regardless of its previous history. Ultimate tensile strength and ductility, on the other hand, showed significant effects of prior exposure. Alloy 80-10-10, after 2000 hr at 450 F, showed no significant change in ultimate strength. Ductility was reduced significantly, by about two thirds, but was probably still adequate. After 10,000 hr at 450 F, the ultimate strength was reduced considerably and the ductility was very low. After 2000 hr at 550 F, the residual tensile properties were very similar to those for the specimens that had been at 450 F for 2000 hr.

For the 85-5-5-5 alloy, neither the 2000- nor the 10,000-hr testing at 450 F produced a significant decrease in room-temperature tensile properties, although the ductility was down a moderate amount. The specimen that had been creep tested at 550 F for 2000 hr had very low tensile properties at room temperature. An oxidized crack was present in the fracture. Apparently, this crack had formed during the creep testing and its presence was the cause of the low room-temperature tensile properties.

The Navy "M" alloy, after it had been creep tested at 450 F for 2000 hr, showed little change in ultimate strength or elongation. Both were decreased slightly. However, at 550 F, both the 2000- and the 10,000-hr creep specimens showed a marked decrease in both strength and ductility, but, on the basis of residual ductility, the useful service life of this alloy was not completely used up.

The following conclusions may be drawn from the room-temperature tensile properties of the various alloys having the prior histories as given in Table IV. Alloy 80-10-10 had poor stability after 10,000 hr at 450 F, and poor stability would also be expected at 550 F if exposed for longer times. With respect to retention of the original tensile properties that the alloy had at room temperature, the 85-5-5-5 alloy had good stability at 450 F for as long as 10,000 hr, but was not suitable for long-time service at 550 F. Navy "M" alloy had adequate stability at 450 F, and some service life remained after 10,000 hr at 550 F.

REFERENCES

(1) "Some Tests on Tin Bronzes at Elevated Temperatures," *Proceedings*, Am. Soc. Testing Mats., Vol. 35, Part II, pp. 205–217 (1935).

(2) Clair Upthegrove and Henry L. Burghoff, "Elevated-Temperature Properties of Coppers and Copper-Base Alloys," Am. Soc. Testing Mats. (1956). (Issued as separate publication *ASTM STP No. 181.*)

JOINT DISCUSSION OF PAPERS BY KURA AND LANG AND BY SIMMONS AND KURA

MR. V. P. WEAVER[1] (*presented in written form*).—Both the paper on "Mechanical and Physical Properties of Three Low-Shrinkage Copper-Base Casting Alloys" and its companion paper "The Creep Properties of Three Low-Shrinkage, Copper-Base Casting Alloys" answer partially a long-felt need in the Subcommittee on Nonferrous Materials of the ASME Boiler and Pressure Vessel Code Committee for reliable data on which to base allowable design stresses.

At the end of the introduction, mention is made that the first paper constitutes the first portion of a continuing program in which additional alloys will be studied. Could the authors at this point disclose any further details on just what will be the additional alloys?

In Fig. 4 of the first paper the dual curves for yield strength at both 0.5 per cent extension under load and 0.2 per cent offset will be of particular value in controversial discussions concerning the merits of the two methods.

MR. D. L. LAVELLE.[2]—The Brass and Bronze Ingot Inst. and the authors are certainly to be commended for the work reported upon in these papers. A statement that appears in the introduction to the first paper is worth emphasizing:

> "Although data on some of the mechanical and physical properties of these alloys are available in the literature, the authenticity of these data is not precisely known."

This is a masterful understatement. It is remarkable that we have come as far as we have in copper-base alloy technology without accurate and reliable determinations of these properties. I would like to point out that some of the properties differ from the published values, and that it would seem well for the ASTM to reevaluate the previous published data.

MR. J. G. KURA (*author*).—Mr. Lavelle's comments are well made. Technology has moved along and it was timely to consider whether the values given in various reference sources were representative of today's technology.

The work that is being reported now is part of a continuing program. Additional commercial alloys are under the same type of investigation. In reply to Mr. Weaver, the next group of alloys comprises two manganese bronzes, ASTM Specifications B 147 – 52, 8A and B 147 – 52, 8C; leaded semi-red brass, ASTM Specification B 145 – 52, 5B; leaded nickel-silver, ASTM Specification B 149 – 52, 11A; and silicon brass, ASTM Specification B 198 – 52, 13B.

MR. L. A. RICE.[3]—I am particularly interested to know whether work is contemplated in a range at somewhat lower temperatures and somewhat lower values of creep. There seems to be an absence of data in the area where creep is incipient. Data would be particularly useful in instrument type applications where creep must be reduced to the vanishing point.

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[1] Metallurgical Engineer, The American Brass Co., Waterbury, Conn.

[2] Research Department, Am. Smelting and Refining Co., South Plainfield, N. J.

[3] Delco-Remy Division, General Motors Corp., Anderson, Ind.

MR. W. F. SIMMONS (*author*).—At the present time the maximum interest appears to be in obtaining data of the type needed by the Boiler Code Committee for the determination of maximum allowable stresses. For this purpose, short-time yield and tensile strength data are needed as well as the creep and rupture data. The data obtained in this investigation indicate that at low temperatures, say below 300 F, the allowable stresses will be determined for the short-time data, and that when the creep stresses are low enough to avoid excessive initial strain the creep rates will be very low. Early in this program, when we were exploring the field to decide at what temperatures these alloys should be evaluated, we made a few creep tests at 250 F, and for all three alloys we found that at stresses below the proportional limit the creep was nil. However, as long as our primary interest is in determining data for Boiler Code use we will be determining creep rates of the order of 0.00001 per cent per hr, one of the criteria of the Boiler Code Committee.

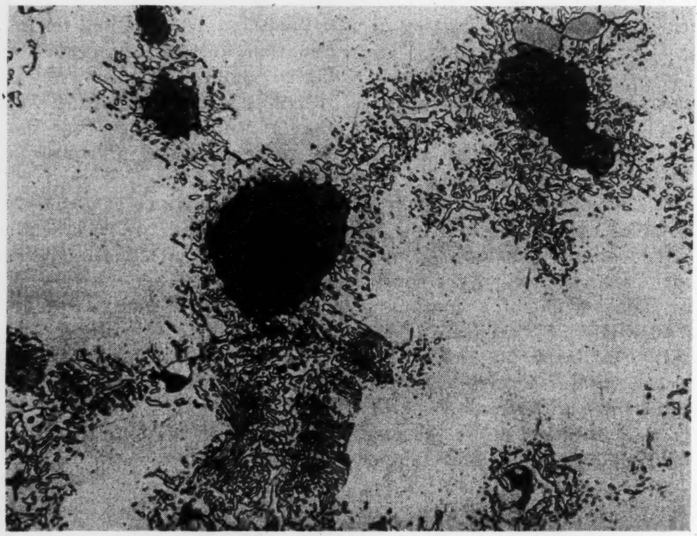

FIG. 1.—Structure of the 80-10-10 Alloy After Creep Testing for 2020 hr at 550 F at a Stress of 5000 psi. Etched in $FeCl_3$-HCl (×500).

MR. J. S. VANICK.[4]—Several years ago Edwards[5] of the Canadian Bureau of Mines reported upon some short-time elevated temperature tests on several tin bronzes. One of them, an "M" metal type, when tested at 650 F developed weakness and low ductility of an order corresponding to the values shown in Table IV for the tests on "M" bronze after the creep test exposure.

In tests on tin-bronzes containing more than 3 per cent of nickel, a low elongation occurs, but the cold strength is substantially increased and the hot strength is almost twice as great as that obtained for Navy "M" metal. These 5 per cent nickel, 5 per cent tin bronzes are precipitation hardening, and their behavior can be accounted for through our belief that a nickel-tin-copper phase is precipitating and lending a hardening and strengthening effect to the heat-exposed material. We have been careful to call attention to this deterioration in ductility. The strength in the nickel-tin bronze is substantially increased, almost doubled.

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[4] Metallurgist, International Nickel Co., Inc., New York, N. Y.

[5] J. O. Edwards, "Short-Time Elevated Temperature Properties of Three Copper-Base Casting Alloys," 59th Annual Meeting, Am. Foundrymen's Soc., May, 1955.

Do the authors have any evidence of microscopic segregation or precipitation that would explain the instability of "M" bronze or depreciation in properties?

MR. KURA.—Metallographic examinations were made to answer Mr. Vanick's questions whether structural changes were observed that could account for the instability of the Navy "M" alloy. Unfortunately, the few bars that had been creep tested for 10,000 hr and subsequently tested at room temperature were discarded. Therefore, other bars that had been tested in creep were examined.

A structural change was found to have occurred to some extent in all three of the alloys tested in creep. When compared with the as-cast structure, a considerable change occurred in the 80-10-10 alloy at 550 F. The structural change was less marked in the Navy "M" alloy at 450 F. Only a small change occurred in the 85-5-5-5 alloy at 550 F.

TABLE I.—STRUCTURAL CHANGES IN ALLOYS TESTED.

Prior History				Microstructure
Alloy	Temperature, deg Fahr	Stress, ksi	Test Duration, hr	
80-10-10		As-cast		Large lead particles and delta dispersed throughout an alpha matrix.
	350	20.0	2022	Some eutectoid structure around the surface of the lead and delta particles.
	550	5.0	2020	A large amount of eutectoid structure and a large amount of precipitated epsilon phase (see Fig. 1).
Navy "M"		As-cast		Small lead particles scattered throughout the alpha matrix and a few particles of delta phase.
	450	13.0	2018	A large amount of eutectoid structure present around the lead particles.
	550	8.0	1176	Considerable amount of eutectoid structure. Fewer unreacted lead and delta particles. Considerable amount of discontinuous epsilon along grain boundaries.
85-5-5-5		As-cast		Lead particles in an alpha matrix with a few delta particles present.
	350	11.0	1800	Same as the as-cast structure.
	550	10.0	744	Very small amount of the eutectoid structure. Some precipitate of epsilon at the grain boundaries.

The photomicrograph in the accompanying Fig. 1 shows the structure of the 80-10-10 alloy after creep testing for 2020 hr at 550 F under a stress of 5000 psi. It is typical of the type of structural change that occurred in all three alloys. The metastable copper-tin matrix has undergone the alpha-epsilon eutectoid reaction to form a eutectoid structure in some areas. Bolton[6] found a similar eutectoid reaction in the 88-10-2 alloy. In other areas of our alloy, particles of epsilon appear to have precipitated during creep testing. The eutectoid reaction occurred at the periphery of the lead, and the edges of the delta particles became jagged. The delta particles became partially or completely consumed. The attack on the lead particles could be (1) a ternary eutectoid reaction, (2) a eutectoid reaction of a system more complex than a ternary, or (3) the reaction of the tin in the matrix with the lead particles. The formation of the eutectoid structure may explain the depreciation of properties since such a structure usually imparts higher strength with some sacrifice in ductility.

TABLE II.—RESULTS OF TENSION TESTS ON UNBROKEN CREEP SPECIMENS.

	Tensile Strength, tons per sq in.	Elongation, per cent
88-10-2 Gunmetal..........	8.0 to 16.2	nil to 6.0
86-7-5-2 Gunmetal........	7.2 to 14.1	nil to 7.0
85-5-5-5 Gunmetal.........	8.6 to 13.3	3.0 to 5.0

A description of the structural changes in some specimens of the three alloys is presented in the accompanying Table I.

The relative amount of eutectoid structure in these three alloys can be correlated with their tin content. The 80-10-10 alloy contains about 9 per cent tin. The as-cast alloy is supersaturated with tin and is metastable. At 350 F and under stress, some eutectoid reaction occurs and part of the delta phase decomposes to alpha and epsilon. At 550 F, considerably more eutectoid reaction has occurred. The Navy "M" contains about 6 per cent tin. It also is metastable but is less supersaturated. Less eutectoid reaction occurred during creep testing. The 85-5-5-5 alloy contained about 4.5 per cent tin. It was near the alpha and alpha-epsilon phase boundary, at 550 F. Therefore, it was stable and no eutectoid reaction occurred except at a few areas in which the tin content may have been slightly higher probably as a result of coring. A few particles of delta, present because of inhomogeneity, could decompose by the eutectoid reaction or go into solution during creep testing. Thus, the amount of eutectoid depends primarily upon the tin content. The nickel, zinc, and lead content undoubtedly shift the equilibrium composition and temperature of the eutectoid.

MESSRS. F. HUDSON[7] AND MANTLE[8] (*by letter*).—In some work in which the British Non-Ferrous Metals Research Assn. and the Mond Nickel Co. are collaborating there appeared to be little difference in creep properties between 86-7-5-2 and 85-5-5-5 gunmetals at temperatures in the range 450 to 550 F. However, our investigations did not indicate that the 86-7-5-2 alloy was more stable at elevated temperatures than 85-5-5-5.

Tension tests conducted on unbroken creep specimens gave the results shown in the accompanying Table II.

While in some cases there was a reduction in tensile strength compared with the average strength before test, practically all the specimens which had been creep tested for long times suffered marked reduction in ductility. All three alloys suffered this reduction to about the same extent.

The conclusions reached in terms of stress to produce 0.1 per cent plastic creep strain in 10,000 hr for the commercially produced materials were as follows:

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[6] J. W. Bolton, "Some Tests on Tin Bronzes at Elevated Temperatures," *Proceedings*, Am. Soc. Testing Mats., Vol. 35, Part II, p. 204 (1935).

[7] Metallurgist, The Mond Nickel Co., Ltd., London (England).

[8] Metallurgist, British Non-Ferrous Metals Research Assn., London (England).

1. At 450 F the material with the highest resistance to creep is the 88-10-2 alloy followed closely by the 85-5-5-5 alloy;
2. At 500 F the material with the highest resistance to creep is the 85-5-5-5 alloy followed by the 86-7-5-2 alloy; and

TABLE III.—CHARPY V IMPACT ENERGY, FT-LB.

Temperature, deg Fahr	85-5-5-5	Navy "M"
210	13	15
80	18	18
−100	13	20
−305	11	18

TABLE IV.—SUPPLEMENTARY RUPTURE DATA FOR 80-10-10, 85-5-5-5, AND NAVY "M" ALLOYS.

Specimen Number	Temperature, deg Fahr	Stress, ksi	Rupture Time, hr	Elongation per cent	Reduction, in Area, per cent	Minimum Creep Rate, per cent per hr
80-10-10 Alloy:						
3462BL	350	27.5	213[a]	13.72[b]	...	0.0010
3462DR	450	27.5	0.9	14.0	16.3	...
3462LR	550	16.0	26.0	2.7	8.6	...
3462BR	550	13.5	145.7	2.2	4.3	0.0062
85-5-5-5 Alloy:						
3460BL	350	25.5	235[a]	13.51[c]	...	0.0020
3460BR	450	28.0	0.5	26.3	14.1	...
3460DL	550	15.5	7.3	7.0	3.4	0.066
Navy "M":						
3459BL	350	35.0	36.0	34.2	34.8	...
3459FR	350	32.0	211[a]	...	...	...
3459CR	450	25.0	13.2	20.8	27.7	...
3459DR	450	21.0	208[a]	10.0[d]	...	0.0016

[a] Test discontinued.
[b] Elongation after 213 hr.
[c] Elongation after 160 hr.
[d] Elongation after 180 hr.

3. At 550 F the material with the highest resistance to creep is again the 85-5-5-5 alloy with little to choose between the other two alloys.

For materials produced under laboratory controlled conditions the creep resistance of the 85-5-5-5 alloy is again greater than that of the 86-7-5-2 alloy at 550 F.

In more general terms, the 85-5-5-5 alloy is considerably more creep resistant than the others at the highest test temperatures and is not much less creep resistant than the others at lower temperatures.

We believe that an alloy in which 3 per cent of nickel replaces equal amounts of tin, zinc, and lead will produce an improved composition with better creep properties than any of the alloys reported upon. An alloy of this type would also be less sensitive to the effect of section thickness and produce stronger castings on a commercial basis.

MR. SIMMONS.—The discussion by Messrs. Hudson and Mantle indicated that they found little difference in the stability of 85-5-5-5 and 86-7-5-2 alloys. Also, we found little difference in the long-time stability of the three alloys tested in this investigation. However, our data give Navy "M" alloy a slight edge strengthwise. This alloy had better rupture properties than 85-5-5-5 alloy at 550 F. The two "rupture" tests of 85-5-5-5 alloy at 550 F, 11,000 and 10,000 psi, started out to be creep tests, but they

TABLE V.—REVISED RESULTS OF CREEP-RUPTURE TESTS.

Alloy	Temperature, deg Fahr	Rupture Strength, ksi	
		100 hr	1000 hr
80-10-10	350	28.9	...
	450	18.8	15.0
	550	13.9	...
85-5-5-5	350	26.6	...
	450	19.0	15.4
	550	12.4	9.6
Navy "M"	350	33.1	...
	450	22.2	...
	550	14.4	11.7

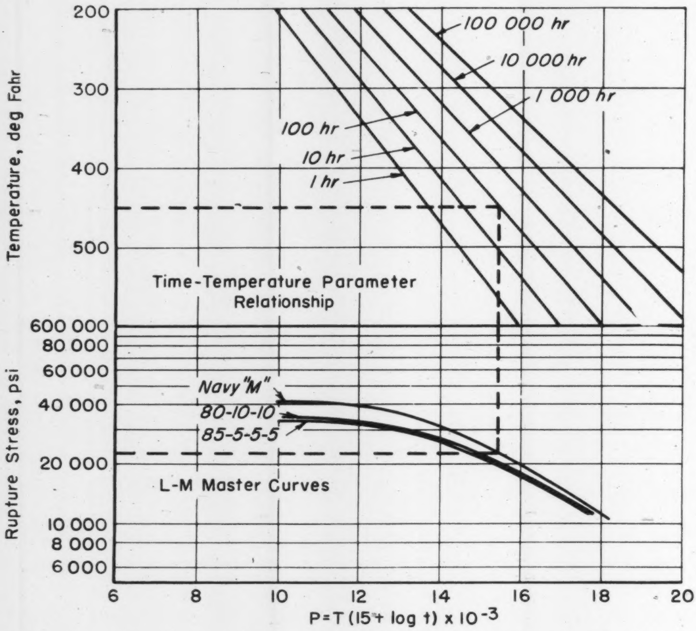

FIG. 2.—Larson-Miller Master Rupture Curves and Time-Temperature Parameter Relationship for Three Cast Copper-Base Alloys.

broke with brittle failures. The creep test of 85-5-5-5 alloy at 550 F and 8000 psi, which ran for 2000 hr, showed an oxidized crack when it was pulled in tension after creep. This crack had formed during creep testing, and undoubtedly would have resulted in premature failure.

On the other hand, none of the Navy "M" alloy specimens tested at 550 F showed "brittle" rupture failures. It should be emphasized that 550 F is a very high temperature for either of these leaded alloys.

I might mention a recently published British article, "Creep Properties of Three Copper Alloys" by J. McKeown and R. D. S. Lushey, *Metal Industry*, Vol. 92, No. 17, pp. 327–329, April 25, 1958. This article includes some data for 85-5-5-5 alloy from cast $\frac{1}{4}$ in.-thick plate. This cast plate has lower strength and ductility than our castings. Their creep rates at 400 F were about the same as ours at 450 F.

MR. E. A. LANGE[9] (*by letter*).—The Charpy V-notch impact properties of two of the alloys discussed in the papers were investigated at the U. S. Naval Research Laboratory to −300 F. It is interesting to note the good agreement between the values obtained in the two investigations. The Charpy V-notch test bars were cut from 1 in. thick plate castings which would have cooling rates comparable to those for the castings used in the subject investigation (accompanying Table III).

The Charpy impact energy values for the two bronzes are low when compared with values for most ferritic steels tested above their transition temperatures. However, these two bronzes do not become notch brittle at low temperatures, and their fracture characteristics are essentially the same over a wide range of low temperatures.

MR. SIMMONS (*author's closure*).—Since the time that this paper was presented we have made several additional creep-rupture tests on all three alloys. The results of these tests are given in the accompanying Table IV. The rupture data from these tests permit a more complete tabulation of the stresses required to produce rupture in 100 or 1000 hr than was given in Table III of the paper. The revised tabulation is given in the accompanying Table V.

One of the purposes in making these additional tests was to obtain a minimum of rupture data to permit an evaluation of the usefulness of the Larson-Miller parameter[10] as applied to these cast copper-base alloys.

The results indicated that the value of C in the parameter $P = T(C + \log t)$ was not constant over the ranges of temperature, stress, and time of interest for these alloys. Therefore, the parameter method did not offer any advantages over the conventional methods of plotting the data when a high degree of precision is desired. However, when a qualitative comparison of the rupture strengths of the various alloys is desired, the Larson-Miller curves offer a real advantage over the conventional methods of handling the data. The accompanying Fig. 2, which contains an approximation of all of the rupture data for all three alloys in a single graph, is an example of this advantage. Figure 2, however, is not recommended as a source of design data.

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[9] Head, Casting Section, Metallurgy Division, U. S. Naval Research Laboratory, Washington, D. C.

[10] F. R. Larson and James Miller, "A Time-Temperature Relationship for Rupture and Creep Stresses," *Transactions*, Am. Soc. Mechanical Engrs., Vol. 74, No. 5, pp. 765–775, July, 1952.

ISOCHRONOUS STRESS-STRAIN CURVES FOR SOME MAGNESIUM ALLOYS SHOWING THE EFFECTS OF VARYING EXPOSURE TIMES ON THEIR CREEP RESISTANCE*

By Robert B. Clapper[1]

Synopsis

The short-time creep resistance of some new elevated-temperature magnesium alloys were studied to show the effect of time (5 sec to 3 hr) at test temperatures prior to testing. Rapid heating methods were used in this investigation, and automatic-recording techniques were employed. For total extensions of 0.5 per cent, the materials given the shorter soaking times of 5 sec are shown to have equal or better short-time creep resistance than those given the 3-hr soaking time. The effect of heating rates on short-time creep resistance is also discussed. The data are presented in the form of isochronous stress-strain curves to facilitate their use in design.

Short-time creep data have assumed importance as design criteria for aircraft and missile structures subjected to aerodynamic heating. Smith (**1**)[2] has used temperature-yield curves to study the effects of heating rates on materials. Kattus and Dotson (**2**) showed limiting creep-strain curves for a given stress by plotting temperature *versus* time after rapid heating. Goldin (**3**) points out that cold-structure instability analyses require only the complete stress-strain curve, but heated structures require the time and temperature parameters which are supplied by isochronous stress-strain curves for the material being studied. Because of the current wide interest in isochronous stress-strain curves, the short-time creep data obtained in this study are presented in this manner.

The isochronous stress-strain curve is developed by: (*a*) plotting the total extensions measured for given times from loading for each stress tested at the test temperature, and (*b*) fairing a curve between the points of constant time. Obviously the strain for any time greater than zero must be at least equal to the elastic strain for any stress level.

It becomes almost necessary to record data autographically for short-time creep testing when: (*a*) heating times to test temperature are less than 1 min, (*b*) soaking times at temperature before loading are 5 sec or less, (*c*) strain readings are required at 5-sec intervals or less, and (*d*) complete stress-strain loading curves are desired. Automated techniques were used extensively in this

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* Presented at the Sixty-first Annual Meeting of the Society, June 22–27, 1958.

[1] Metallurgical Laboratory, The Dow Chemical Co., Midland, Mich.

[2] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 825.

TABLE I.—SPECTROGRAPHIC ANALYSES AND SHORT-TIME TENSILE PROPERTIES IN THE LONGITUDINAL DIRECTION OF THE MAGNESIUM ALLOYS.

Chemical Composition, per cent		Mechanical Properties[a]			
		Test Temperature, deg Fahr	Elongation,[b] per cent	Tensile Yield Strength, psi	Tensile Strength, psi
HK31A-H24 SHEET					
Aluminum	<0.03	80	14.0	27 500	35 500
Calcium	<0.01	300	20.5	23 400	25 500
Copper	<0.005	400	26.5	20 700	22 200
Iron	0.003	500	25.0	17 400	19 500
Manganese	0.048	600	37.5	6 200	13 500
Nickel	<0.001	...	...	...	...
Lead	<0.001	...	...	...	...
Silicon	<0.01	...	...	...	...
Tin	<0.01	...	...	...	...
Zinc	<0.02	...	...	...	...
Thorium	2.6	...	...	...	...
Zirconium	0.92	...	...	...	...
Magnesium	balance	...	...	...	...
HM21A-T8 SHEET					
Aluminum	<0.03	80	7.5	23 500	34 300
Calcium	<0.01	300	19.5	20 600	23 500
Copper	<0.005	400	26.0	17 500	19 400
Iron	0.001	500	23.5	15 700	17 200
Manganese	0.58	600	17.5	12 800	15 000
Nickel	<0.001	700	29.5	6 800	10 700
Lead	<0.001	800	42.5	2 700	5 100
Silicon	<0.01	900	73.0	800	1 800
Tin	<0.01	...	...	...	...
Zinc	<0.02	...	...	...	...
Thorium	2.25	...	...	...	...
Magnesium	balance	...	...	...	...
HM31XA-F EXTRUSION					
Aluminum	<0.03	80	3.0	31 500	37 800
Calcium	<0.01	300	...	24 000	25 800
Copper	0.002	400	20.0	21 600	22 600
Iron	<0.001	500	22.0	19 600	20 000
Manganese	1.78	600	27.0	16 500	17 600
Nickel	<0.001	700	55.0	9 000	10 700
Lead	<0.001	800	...	3 700	4 800
Silicon	<0.01	900	66.0	1 400	2 100
Tin	<0.01	...	...	....	...
Zinc	0.15	...	...	....	...
Thorium	3.58	...	...	....	...
Magnesium	balance	...	...	....	...

[a] 1. All elevated test temperatures were attained in less than 1 min by resistance heating.
2. Strain rate to tensile yield strength was 0.005 per min.
3. Strain rate beyond tensile yield strength was 0.1 per min.
4. Specimens were machined in accordance with ASTM Method E 8 – 57 T,[3] Fig. 6.

[b] Elongation was measured over 2 in. for room-temperature tests and over 1 in. for elevated-temperature tests.

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[3] Tentative Methods of Tension Testing of Metallic Materials (E 8 – 57 T), 1958 Book of ASTM Standards, Part 3.

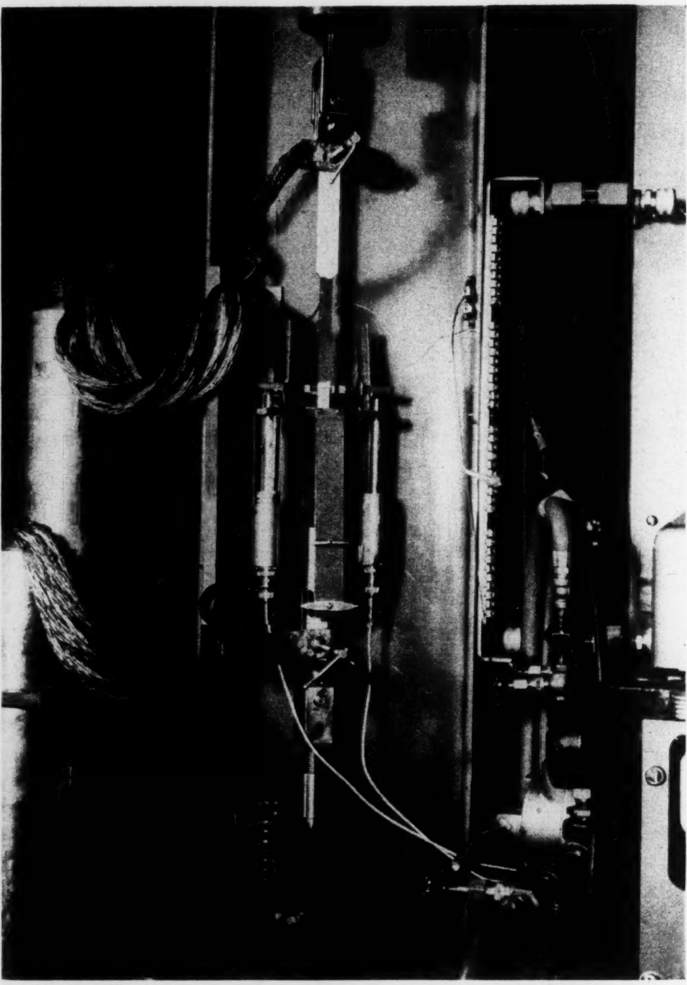

FIG. 1.—Detail Arrangement of Specimen with Extensometer, Electrical Connections, and Thermocouples.

study. Additional benefits of automation were: (*a*) correlation of autographic temperature and strain data *versus* time, (*b*) complete autographic temperature-gradient data, (*c*) autographic data for the accurate determination of loading or straining rates, and (*d*) autographic data for the determination of heating rates.

The effects of rapid heating and of soaking times on the short-time creep properties of three magnesium-thorium alloys designed for elevated-temperature service are reported in this paper.

TEST MATERIALS

The HK31A-H24 and HM21A-T8 sheet alloys (0.064 in.) were tested in the longitudinal direction. HK31A is a magnesium-thorium alloy containing zirconium, and HM21A is a magnesium-thorium alloy containing manganese. HM31XA-F extruded tubing (10 in. outside diameter by 0.315 in. wall thickness), which was also tested, is a magnesium-thorium alloy containing manganese. The temper designations are in accordance with ASTM Recommended Practice B 296.[4] The chemical composition and mechanical properties (Table I) meet specification requirements for commercial materials. The extruded tubing properties are also representative of commonly extruded bars, rods, and shapes of this alloy.

All specimens tested for short-time creep were flat with over-all dimensions 24 in. in length by $\frac{7}{8}$ in. in width. The reduced section was approximately 11 in. in length by 0.515 in. in width, with a 2-in. long standard 0.505-in. width in the middle of the reduced section where the extensometer was mounted.

The extruded specimens were milled to a 0.125-in. thickness from the middle of the wall thickness.

APPARATUS AND TESTING PROCEDURE

A creep-rupture testing machine of 12,000-lb capacity (Riehle model CR-12) converted to a 10 to 1 lever ratio was used in this program. It was equipped with special spherical ball-seat joints, and button-head adaptor rods with split washers for quick disconnecting. Special flat sheet grips (Riehle No. 328-49B), coupled with the 24-in. long specimen, aided considerably in minimizing alignment problems.

The extensometer (1-in. gage length) used two independently measuring linear variable-permeance type transducers (Crescent Engineering model LC-2-2.0). The transducers were linear for displacements to 0.2 in. and were sensitive and accurate to $<25\ \mu$ in. Their outputs were averaged electrically. The details of the specimen and extensometer arrangement are shown in Fig. 1.

A stress-strain recorder (Moseley X-Y, model 3 Autograf) was used to obtain the stress-strain curve resulting from the loading of the specimen. The averaged strain signal was amplified by a controller-amplifier (Crescent model EKA-2D) prior to being measured on one axis of the stress-strain recorder. The same strain signal was also measured by one function of a two-function-time recorder (Brown $X_1 - X_2$, model SC153X (27)-(VP)-II-(W72)-(30)-(V)) connected in parallel with the stress-strain recorder.

Stress for obtaining a stress-strain curve was obtained directly by recording stress on one axis of the stress-strain recorder. This was accomplished by controlling the excitation voltage of an SR-4 load cell which was in series with the specimen. A plot in terms of stress resulted by regulating the voltage with a linear potentiometer in a circuit designed so that a duo-dial on the potentiometer shaft could be read directly in units of area.

Loading was accomplished by remote control of an air-cylinder under the bale pan. It was capable of loading the specimen in less than 1 sec. The loading was generally accomplished in approximately 2 sec. However, loading times varied from 1 to 4 sec, depending on stress level and temperature used. Translated into terms of straining rate, values of 0.020 to 0.090 per min were obtained.

To study the effects of heating rate and strain rate, it was necessary to control the loading rates more precisely. Limited calibration tests of the air-cylinder control valve resulted in strain-rate control to approximately ±0.005 per min. This variation in strain rate is considered to have a negligible effect on the data based on a study by Fenn and Gusack (**4**).

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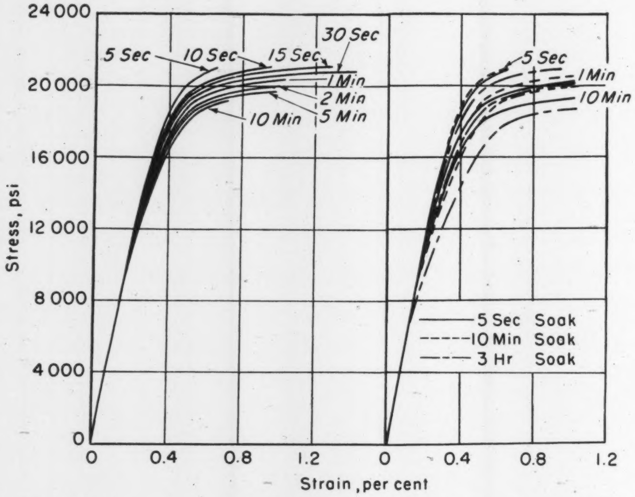
[4] Recommended Practice for Temper Designation of Light Metals and Alloys, Cast and Wrought (B 296 - 56), 1958 Book of ASTM Standards, Part 2.

Specimens were heated by resistance heating for all tests. Current was supplied by a 440-v to 0.8 to 3.0-v step-down transformer, and regulated by a Westinghouse Furnatron Control system (FST-2) equipped with a Leeds & Northrup controller (series 60). A 30-gage iron-constantan thermocouple, percussion welded to the center of the gage length of the specimen with a capacitor-discharge welder, was used to supply a temperature signal to a recording controller.



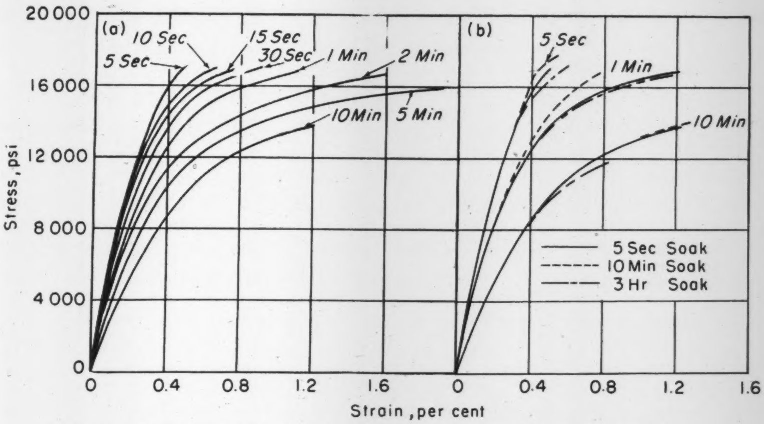
(*a*) Rapid heating with 5-sec soaking time. (*b*) Effect of soaking time.

Fig. 2.—Isochronous Stress-Strain Curves for HK31A-H24 0.064-in. Sheet at 400 F.

The heating time to test temperature varied from approximately 10 to 30 sec, depending on test temperature and thickness of the specimen. Exposure time was measured from the time the temperature of the control thermocouple was within 3 F of the test temperature.

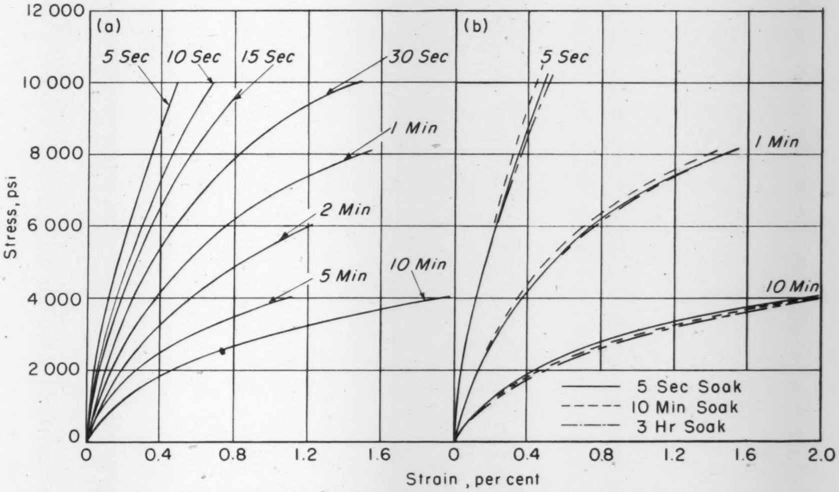
Resistance heating of the specimen to test temperature was used to obtain the shorter soaking times of 5 sec to 10 min prior to testing, while the specimen was mounted on the machine. The material soaked for 3 hr was soaked in an ordinary electric furnace with thermocouples attached to measure actual specimen temperature, which was controlled to less than ±5 F variation from testing temperature. The specimens required approximately 10 to 15 min to get to temperature and were air cooled prior to installing in the creep machine. From this point the procedure for testing was the same as was used for the material given the shorter soaking times.

The resistance-heating controller limited temperature over-shoot and fluctuations to ±2 F at the center of



(*a*) Rapid heating with 5-sec soaking time. (*b*) Effect of soaking time.

FIG. 3.—Isochronous Stress-Strain Curves for HK31A-H24 0.064-in. Sheet at 500 F.



(*a*) Rapid heating with 5-sec soaking time. (*b*) Effect of soaking time.

FIG. 4.—Isochronous Stress-Strain Curves for HK31-H24 0.064-in. Sheet at 600 F.

the gage length. Iron-constantan thermocouples were welded at the top and bottom of the gage length to measure thermal gradients. Temperatures at the top and bottom of the gage length were 4 to 8 F lower than those at the center, and varied as a function of test temperature, test time, and specimen thickness. All temperature measurements were continuously recorded. To aid in control of the thermal gradients it was found helpful to paint the specimen outside the gage length with carbon black to promote heat emission, since this area tended to overheat. The gage collars also aided in control of thermal gradients by acting as heat sinks in the area of their attachment.

Testing procedures followed the practices outlined by the Aircraft Industries Assn. of America, Inc., under Standard Test Procedure ARTC-13-T-2 for Short-Time Tensile-Creep and Rupture Testing of Metallic Materials, and ASTM Recommended Practice E-21.[5]

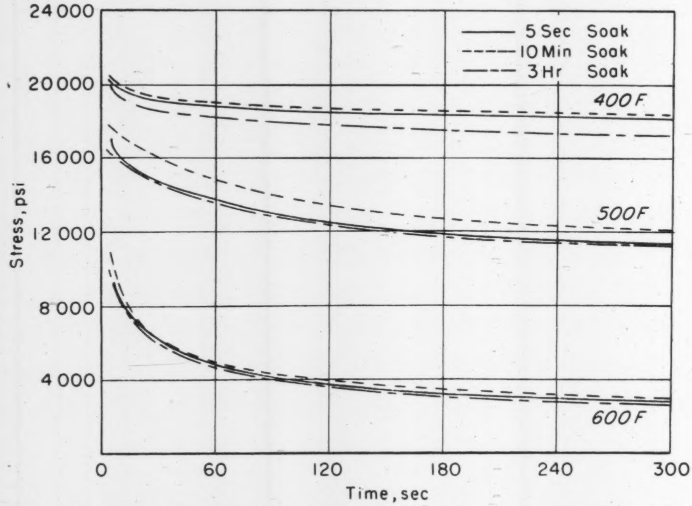


FIG. 5.—Effect of Soaking Time on the Short-Time Creep Strength for HK31A-H24 0.064-in. Sheet at 0.5 per cent Total Extension.

RESULTS AND DISCUSSION

All materials were tested for short-time creep: (1) for 10 min or (2) to a maximum total extension of 2 per cent, whichever occurred first. In all cases total extension is the only strain shown. Thermal expansion effects are not included in these isochronous data. The data shown in this paper represent the results of testing one lot of material only, and cannot necessarily be considered as typical for these alloys. Test time, as used herein, is defined as the sum of the loading time plus the creep time (or time to total extension). The use of these test times is illustrated in Fig. 2(*a*).

The purpose of this study was to determine the short-time creep characteristics of three new elevated-temperature magnesium alloys under rapid heating conditions and to show the effect of soaking time (5 sec, 10 min, and 3 hr) at the test temperatures prior to testing on their creep resistance. Detailed isochronous stress-strain curves for the 5-sec soaking time are presented for each alloy tested, for each test temperature, and for the test times of 5 sec to 10 min. HK31A-H24 sheet was tested at 400, 500, and 600 F (see Figs. 2(*a*), 3(*a*) and 4(*a*). HM21A-T8 sheet was tested at 600, 700, and 800 F (see Figs. 6(*a*), 7(*a*), and 8(*a*)). HM31XA-F extruded

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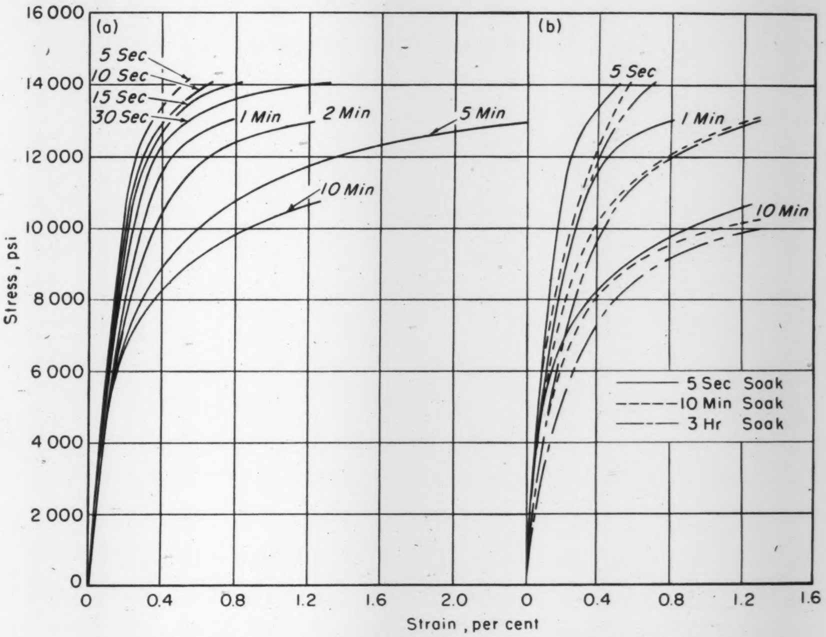
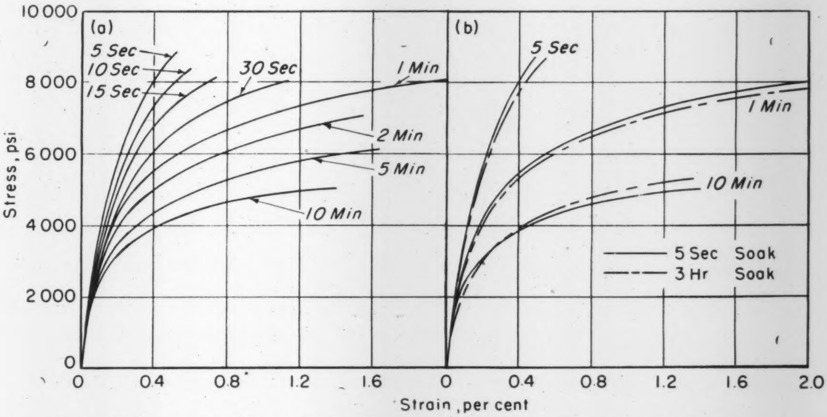
[5] Recommended Practice for Short-Time Elevated-Temperature Tension Tests of Metallic Materials (E 21 – 58 T), 1958 Book of ASTM Standards, Part 3.



(*a*) Rapid heating with 5-sec soaking time. (*b*) Effect of soaking time.

FIG. 6.—Isochronous Stress-Strain Curves for HM21A-T8 0.064-in. Sheet at 600 F.



(*a*) Rapid heating with 5-sec soaking time. (*b*) Effect of soaking time.

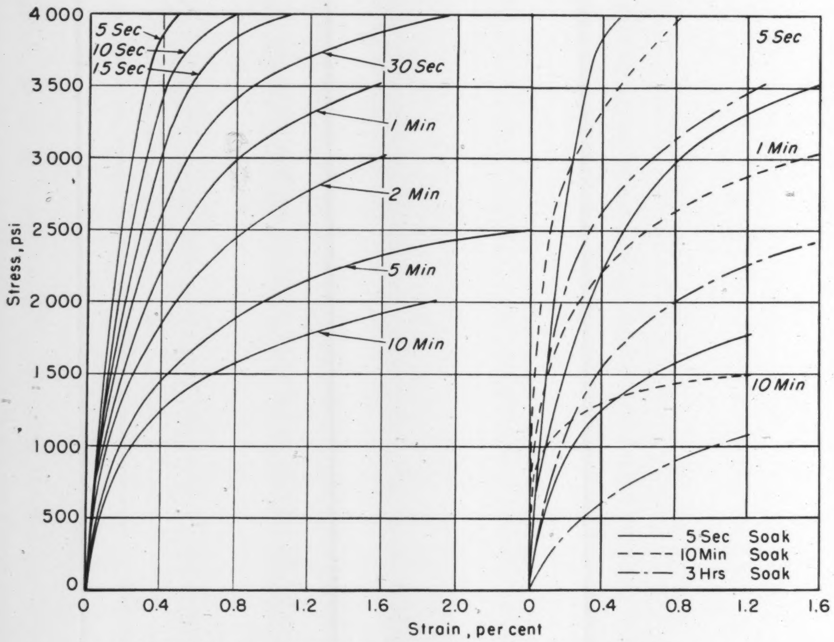
FIG. 7.—Isochronous Stress-Strain Curves for HM21-T8 0.064-in. Sheet at 700 F.

tubing was tested at 600, 700, and 800 F (see Figs. 11(*a*), 12(*a*) and 13(*a*)).

PRINTS AND DISCUSSION

HK31A-H24 Sheet:

The effects of soaking time on the isochronous stress-strain curves of HK31A-H24 sheet material are shown for selected test times in Figs. 2(*b*), 3(*b*) and 4(*b*). The effects of both soaking time and test time on the limiting creep stress (0.5 per cent total extension) are shown in Fig. 5 for the temperatures tested. The material given the 10-min soak appeared to have superior creep resistance to the material soaked for 5 sec. Although the material given a 10-min soak is significantly superior to the 5-sec soak at 500 F, it is only marginally better at 400 and 600 F (see Fig. 5) and may be equal when considering the experimental scatter. The material given a 10-min soak is also significantly superior to that given a 3 hr soak at both 400 and 500 F, and again is only marginally better than at 600 F.



(*a*) Rapid heating with 5-sec soaking time. (*b*) Effect of soaking time.

FIG. 8.—Isochronous Stress-Strain Curves for HM21A-T8 0.064-in. Sheet at 800 F.

HM21A-T8 Sheet:

The effects of soaking times on the isochronous stress-strain curves of HM21A-T8 sheet material are shown for selected test times in Figs. 6(*b*), 7(*b*), and 8(*b*). The effect of both soaking time and test time on the limiting creep stress (0.5 per cent total extension) is shown in Fig. 9 for the temperatures tested. The material given the 5-sec soak appeared to have significantly superior creep resistance to the material soaked for 3

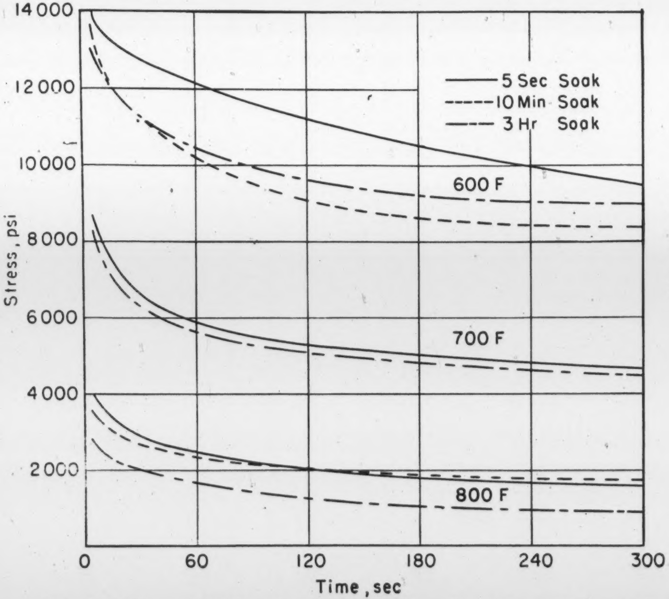


FIG. 9.—Effect of Soaking Time on the Short-Time Creep Strength for HM21A-T8 0.064-in. Sheet at 0.5 per cent Total Extension.

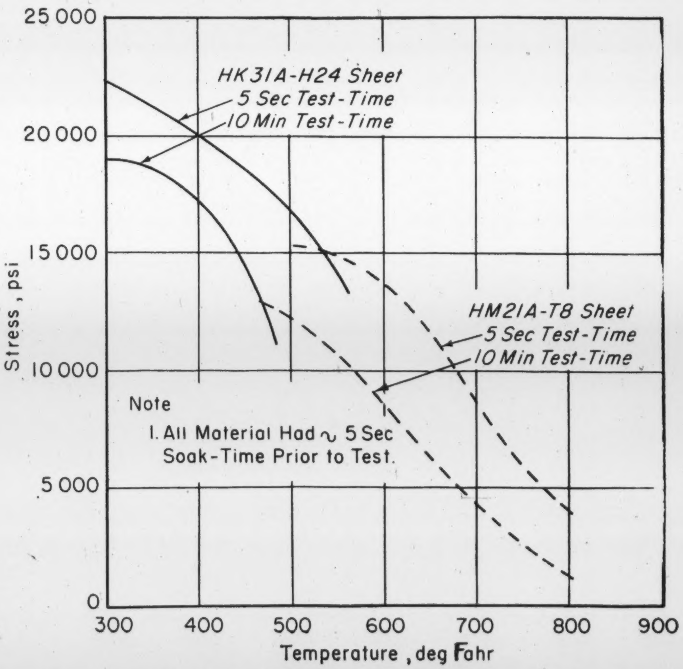


FIG. 10.—Rapid Heating Isochronous Stress-Strain Summary Curves for 0.5 per cent Total Extension Comparing HK31A-H24 and HM21A-T8 Sheet.

hr at 600 and 800 F (see Figs. 6(*b*), 8(*b*), and 9). However, it is only marginally superior at 700 F (see Figs. 7(*b*), and 9) and it may be equal when considering experimental scatter. The material given the 5-sec soak is significantly superior to the material soaked for 10 min at 600 F, (see Fig. 6(*b*)), and is approximately equal to 800 F (see Figs. 8(*b*) and 9). The 500-psi difference at 800 F may be considered within the experimental scatter. A comparison of HM21A-T8 to HK31A-H24 sheet alloys is made by use of the short-time creep strength summary data as shown in Fig. 10. HM21A-T8 sheet has superior creep strengths for the 5-sec test time above 575 F, and for the 10 min test time above 475 F.

HM31XA-F Extruded Tubing:

The effects of soaking time on the isochronous stress - strain curves of HM31XA-F extruded tubing are shown for selected test times in Figs. 11(*b*), 12(*b*) and 13(*b*). The effects of both soaking time and test time on the limiting creep stress (0.5 per cent total extension) are shown in Fig. 14 for the temperatures tested. A comparison of material soaked for 5 sec and 3 hr shows the material soaked for 5-sec to be increasingly superior in creep resistance with increasing test time at all temperatures tested (see Fig. 14). However, the material tested at 700 F does not exhibit differences as large as are shown for the 600 and 800 F test temperatures.

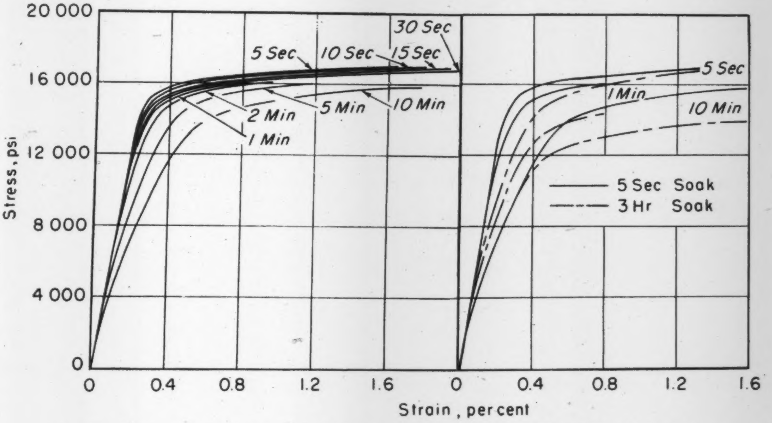
Heating Rate and Strain Rate During Loading:

Heating rate to test temperature and strain rate during loading were calculated for all tests. Tests were replicated, where time and material permitted, on HM21A-T8 and HK31A-H24 sheet alloys. No retests were made on HM31XA-F extruded tubing due to limited material. The initial portions of actual strain-time records from tests of HK31A-H24 sheet at 500 F and 16,000 psi are shown in Fig. 15. The degrees of reproducibility for duplicate specimens after 5-sec, 10-min, and 3 hr soaking times are shown as in curves *A* and *B*, *C* and *D* and *E* and *F*, respectively, for approximately constant strain rates and certain variations in heating rate which were encountered. The differences shown between these curves for duplicate tests are considered relatively small, and not to affect significantly the resulting plots of the isochronous stress-strain curves. Variations of a similar degree were noted for 400 and 600 F tests.

HK31A-H24 sheet does not appear to be significantly affected by varying heating rate from 28 to 140 F per sec (at 500 F) nor by varying the strain rate from 0.030 to 0.065 per min (at 600 F). HM21A-T8 sheet does not appear to be significantly affected by varying strain rate from 0.068 to 0.096 per min (at 700 F). Data were not available for HM21A-T8 sheet to study the effect of heating rate variations, since all replicated tests of this alloy had approximately equal heating rates.

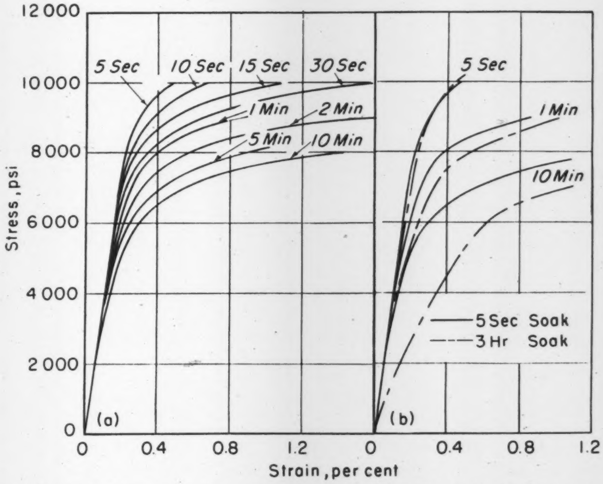
CONCLUSIONS

Short-time creep resistance of the alloys studied, using rapid heating, are significantly affected by: (*a*) soaking time at test temperature prior to testing, (*b*) test temperature, and (*c*) test time. Major differences in the short-time creep resistance were noted between the alloys tested. Neither heating rate nor rate of straining (within the range tested) has a significant effect on the 400, 500, and 600 F short-time creep resistance of HK31A-H24 sheet alloy. Similarly, strain-rate variations (within the range tested) do not have a signifi-



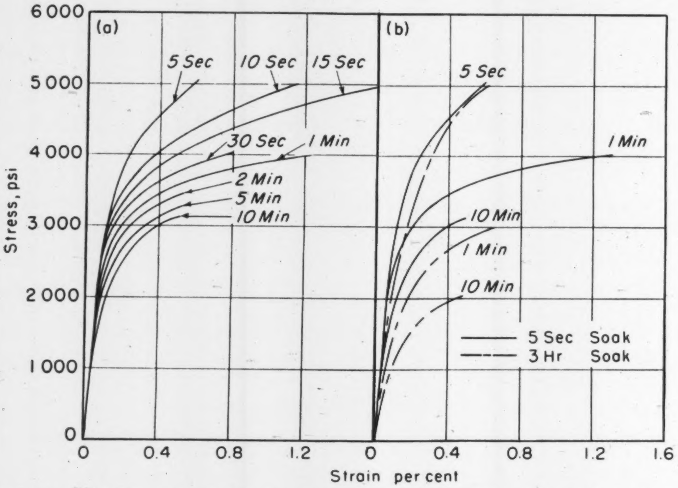
(*a*) Rapid heating with 5-sec soaking time. (*b*) Effect of soaking time.

FIG. 11.—Isochronous Stress-Strain Curves for HM31XA-F Extruded Tubing at 600 F.



(*a*) Rapid heating with 5-sec soaking time. (*b*) Effect of soaking time.

FIG. 12.—Isochronous Stress-Strain Curves for HM31XA-F Extruded Tubing at 700 F.



(*a*) Rapid heating with 5-sec soaking time. (*b*) Effect of soaking time.

FIG. 13.—Isochronous Stress-Strain Curves for HM31XA-F Extruded Tubing at 800 F.

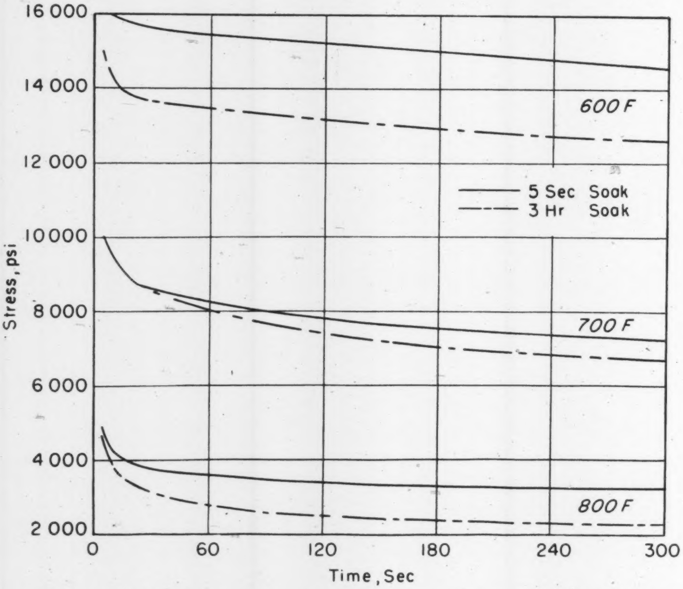


FIG. 14.—Effect of Soaking Time on the Short-Time Creep Strength for HM31XA-F Extruded Tubing at 0.5 per cent Total Extension.

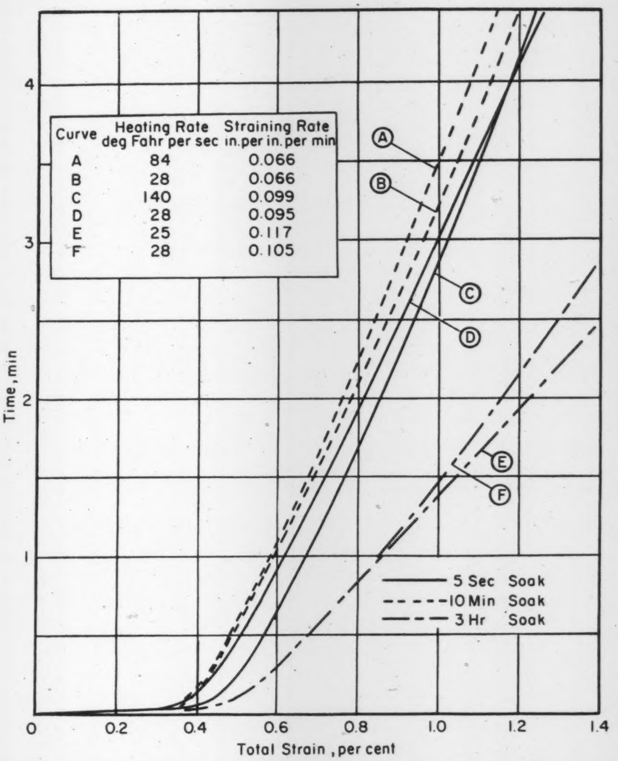


FIG. 15.—Strain-Time Record for Short-Time Creep Tests of HK31A-H24 Sheet at 500 F and 16,000-psi Stress.

cant effect on the 700 F short-time creep resistance of HM21A-T8 sheet alloy. Short-time creep testing procedures for using rapid heating should be standardized to enable direct comparisons of design data between different materials and between different research laboratories.

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(4) R. W. Fenn, Jr., and J. A. Gusack, "Effect of Strain Rate and Temperature on the Strength of Magnesium Alloys," *Proceedings* Am. Soc. Testing Mats., Vol. 58, p. 685 (1958).

YOUNG'S MODULUS OF MAGNESIUM ALLOYS AS A FUNCTION OF TEMPERATURE AND METALLURGICAL VARIABLES*

By Raymond W. Fenn, Jr.[1]

Synopsis

Accurate values for the relaxed Young's modulus, determined on several commercial and experimental magnesium alloys at elevated temperatures by static tension measurements, show significant variations in this important design property. These variations are correlated with differences in alloy content, grain size, prior cold work, and heat treatment. These data are intended to provide information for design use in deflection and buckling calculations and to show the degree of control which the metallurgist has over the relaxed Young's modulus in these alloys.

The use of magnesium alloys at elevated temperatures has revealed a need for accurate values of Young's modulus for use in buckling and stiffness analyses. It has further required a better understanding of the metallurgical factors controlling the relaxed Young's modulus of these alloys to permit the development and production of alloys having the desired modulus values at elevated temperatures. This study was undertaken to provide accurate values of the relaxed Young's modulus on several commercial magnesium alloys over a wide range of temperature and to determine the effect of metallurgical variables such as composition, changes in grain size, cold work, heat treatment, and process history on the relaxed Young's modulus at various temperatures.

Test Materials

The alloys studied are listed in Table I along with their analyses, intended environment (that is, elevated-temperature or room-temperature[2] use), and applicable material specification. The fabrication process and form are shown in Table II with the test results. To facilitate description of the alloys, ASTM nomenclatures (B 275 – 55[3] and B 296 – 56[4] for codification and temper designation of light metals and alloys) are used to describe both commercial and experimental alloys.

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* Presented at the Sixty-first Annual Meeting of the Society, June 22-27, 1958.

[1] Chief, Testing and Instrumentation Section, Metallurgical Laboratory, The Dow Chemical Co., Midland, Mich.

[2] Room-temperature as used in this study does not imply any specific limitation on service temperatures, but is used to distinguish those alloys not specifically designed for elevated-temperature applications.

[3] Recommended Practice for Codification of Light Metals and Alloys, Cast and Wrought (B 275 – 58), 1958 Book of ASTM Standards, Part 2.

[4] Recommended Practice for Temper Designation of Light Metals and Alloys, Cast and Wrought (B 296 – 56), 1958 Book of ASTM Standards, Part 2.

EQUIPMENT AND TEST METHOD

The testing equipment, previously described in detail (**1**)[5] utilizes (1) an overhead lever having a no-load sensitivity of 9.2 g at the specimen, (2) a modified Marten's type optical extensometer having an 8-in. gage length and strain measuring accuracy of 1.6 microinches per in., and (3) adjustable universal joints at the ends of the specimens to permit alignment of the stress and specimen axes.

Three tests were made at 78 F on each specimen within the elastic range to check the alignment of the stress and specimen axes. The specimen was then furnace heated in place to test temperature in approximately 3 hr and again tested three times. The time required to obtain each set of load-strain data was 3 min, and 1 min was required between tests to check the temperature along the gage length of the specimen. If the average of the specimen temperatures changed more than 0.3 F, the data were considered unsatisfactory and the tests were repeated.

The unbiased estimate of standard deviation of the 78 F Young's modulus, based on a triplicate average, generally varied between 0.052×10^6 and 0.061×10^6 psi, while at 600 F it increased to values on the order of 0.07×10^6 to 0.16×10^6 psi.

RESULTS AND DISCUSSION

Differences in anelastic deformation (time-dependent elastic strain) can be a major source of the differences between elastic moduli determined by various test methods and also between alloys within the same alloy system. Kê (**2,3**) has shown anelastic behavior to be a function of grain size, test temperature, and test frequency. Siefert and Worrell (**4**) have considered annealing twins to be another source of anelastic effects. For a theoretical interpretation of these and other factors affecting anelastic behavior the reader is referred to the work of Zener (**5**).

A static tension test method giving relaxed Young's modulus (rather than the unrelaxed modulus) was selected so as to include anelastic deformation in the strain measurements, thereby making the results more useful for design purposes involving static loads. A test temperature of 600 F was usually used to study the effect of metallurgical variables, since the anelastic effects are more pronounced at elevated temperatures, thus making the effects easier to define.

The variations in relaxed Young's modulus of magnesium alloys are generally small at room temperature as shown in Table II (in agreement with the literature review of Mack (**6**) on other metals). However two exceptions are the moduli of high-purity sublimed magnesium (5.8×10^6 psi) and K1X1 alloy (5.0×10^6 psi), which exhibit lower than usual moduli. Considerable experimental difficulty was encountered in attempting to obtain reproducible moduli for sublimed magnesium, particularly at elevated temperatures, where scatter was on the order of 1×10^6 psi. Although the reason for this could not be ascertained, nonuniform grain size and the maximum permissible working stresses may be contributing factors.

EFFECT OF COMPOSITION

Comparisons of the 600 F moduli (Table II) of the elevated-temperature magnesium alloys (containing 1 to 3 per cent thorium or rare earths) and room-temperature magnesium alloys (containing aluminum and zinc) to that of high-purity magnesium show that addi-

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[5] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 837.

TABLE I.—ANALYSES OF MAGNESIUM ALLOYS.

Spectrographic Analyses Except Where Noted.[a]

Alloy Number	Alloy	Applicable Specifications			Composition, per cent												
		Military	ASTM	AMS[f]	Aluminum	Calcium	Copper	Iron	Manganese	Nickel	Lead	Silicon	Tin	Zinc	Zirconium	Thorium	Total Rare Earths
ELEVATED-TEMPERATURE ALLOYS																	
1...	HM21A-T8	...	...	...	<0.03	<0.01	0.003	0.001	0.55[a]	<0.001	<0.001	<0.01	<0.01	<0.02	...	2.41[a]	...
2...	HK31A-H24	MIL-M-26075	B90[c]	4385	<0.03	<0.01	0.003	0.001	0.032	<0.001	<0.001	<0.01	<0.01	<0.02	0.62[a]	2.77[a]	...
3...	HK31A-H24	MIL-M-26075	B90	4385	<0.03	<0.01	0.003	0.002	0.058	<0.001	<0.001	<0.01	<0.01	<0.02	0.71[a]	3.29[a]	...
4...	HK31A-O	MIL-M-26075	B90	4384	0.0003	<0.01	0.0011	0.0008	0.026	0.0004	0.0004	<0.001	<0.001	<0.02	0.65[a]	2.85[a]	...
5...	HK31A-O	MIL-M-26075	B90	4384	<0.03	<0.01	0.003	0.002	0.052	<0.001	<0.001	<0.01	<0.01	<0.02	0.80	2.70	...
6...	HK31A-T6	...	...	...	<0.03	<0.01	0.003	0.001	0.032	<0.001	<0.001	<0.01	<0.01	<0.02	0.62[a]	2.77[a]	...
7...	HM31XA-F	...	...	...	<0.03	<0.01	0.012	0.023	1.67[a]	<0.001	<0.001	<0.01	<0.01	<0.02	...	3.11[a]	...
8...	HM31XA-F	...	...	...	<0.03	<0.01	0.012	0.023	1.92[a]	<0.001	0.001	<0.01	<0.01	<0.02	<0.01	2.44[a]	...
9...	HM21A-T5	...	...	...	<0.005	<0.01	<0.001	<0.001	0.42	<0.005	<0.002	<0.01	<0.01	<0.05	...	2.15	...
10...	HZ32A-T5	QQ-M-56	B80[d]	4447	<0.03	<0.01	<0.005	<0.001	0.045	<0.001	<0.001	<0.01	<0.01	2.34[a]	0.68[a]	3.29[a]	...
11...	HK31A-T6	QQ-M-56	B80	4445	<0.03	...	<0.001	0.0015	0.077	<0.001	0.002	<0.01	<0.01	<0.02	0.69[a]	2.68[a]	...
12...	EK30A-T6	...	B80	...	<0.03	<0.01	0.005	0.002	0.065	0.004	0.007	<0.01	<0.01	<0.02	0.40[a]	...	3.24[a]
13...	EK41A-T6	...	B80	4441	<0.03	<0.01	<0.001	0.0012	0.034	<0.001	0.002	<0.01	<0.01	<0.02	0.49[a]	...	3.87[a]
14...	EZ33A-T5	QQ-M-56	B80	4442	<0.03	<0.01	<0.001	0.0015	0.039	<0.001	0.004	<0.01	<0.01	2.41[a]	0.60[a]	...	2.86[a]
15...	ZH62A-T5	QQ-M-56	B80	...	<0.03	<0.01	<0.001	0.002	0.035	<0.001	0.013	<0.01	<0.01	5.7[a]	0.84[a]	1.93[a]	...
16...	HM11XA-F	...	...	...	<0.03	<0.01	<0.002	0.005	1.49	<0.001	<0.001	<0.01	<0.01	...	...	1.30	...
ROOM-TEMPERATURE ALLOYS																	
17...	AZ31A-H24	QQ-M-44	B90	4376	3.0	0.137	0.006	0.0018	0.55	0.0007	0.003	<0.01	<0.01	0.88	...	...	...
18...	AZ31A-O	QQ-M-44	B90	4375	2.6	0.132	0.009	0.0013	0.58	0.0007	0.003	<0.01	<0.01	0.94	...	...	...
19...	AZ81A-T4	QQ-M-56	B80	...	7.86[a]	<0.01	0.001	0.039	0.25	<0.001	0.003	0.007	<0.01	0.82[a]	...	...	...
20...	AZ91C-T6	QQ-M-56	B80	...	8.75[a]	<0.01	0.003	0.022	0.26	<0.001	<0.001	<0.01	<0.01	0.77[a]	...	...	...
21...	AZ92A-T6	QQ-M-56	B80	4434	9.34[a]	<0.01	<0.001	0.010	0.34	<0.001	0.005	0.008	<0.01	1.88[a]	...	...	...
22...	K1X1-F	...	...	...	<0.03	<0.01	0.002	0.002	0.040	<0.001	<0.001	<0.01	<0.01	<0.02	0.49	...	...
23...	K1X1-F	...	...	...	<0.03	<0.01	0.002	<0.001	0.055	<0.001	<0.001	<0.01	<0.01	<0.02	0.39	...	...
24...	ZK60A-F[b]	QQ-M-31	B107[e]	4352	...	...	...	...	...	...	...	...	...	5.8[b]	0.55[b]	...	...
25...	(P) ZK60B-F[b]	...	...	...	...	...	...	...	...	...	...	...	...	5.5[b]	0.55[b]	...	...

26...	Sublimed magnesium	...	...	...	<0.0001	0.001	0.0001	0.0006	0.0002	<0.0003	0.0005	<0.001	<0.001	<0.01	...	...	...
27...	Electrolytic magnesium	...	...	...	0.0003	<0.01	0.0009	0.031	0.052	0.0002	<0.0004	<0.001	<0.001	<0.02	...	...	...
28...	AZ31-T2	...	...	...	3.04[a]	<0.01	0.003	0.025	0.31	<0.0005	<0.001	0.01	<0.01	1.05[a]	...	...	...
29...	E2-T6	...	...	...	0.002	<0.01	0.004	0.011	0.0009	<0.0003	0.0007	0.006	<0.001	<0.02	...	...	1.65[a]
30...	E4-T6	...	...	...	<0.001	<0.01	0.011	0.021	0.0014	<0.0003	0.0006	<0.001	<0.001	<0.02	...	...	4.37[a]
31...	EM40-T6	...	...	...	0.21	<0.01	<0.001	0.001	0.19	0.001	<0.001	<0.01	<0.01	<0.02	...	...	3.89[a]
32...	ZE10-H24	...	...	...	0.0025	<0.01	0.0006	0.0024	0.019	0.0002	0.0020	<0.001	<0.001	0.84	...	...	0.21[a]
33...	ZE10-O	...	...	...	0.0025	<0.01	0.0006	0.0024	0.019	0.0002	0.0020	<0.001	<0.001	0.84	...	...	0.21[a]

[a] Chemical analysis.

[b] Nominal compositions.

[c] Specification for Magnesium-Base Alloy Sheet and Plate (B 90-58), 1958 Book of ASTM Standards, Part 2.

[d] Specification for Magnesium-Base Alloy Sand-Castings (B 80-58), 1958 Book of ASTM Standards, Part 2.

[e] Specification for Magnesium-Base Alloy Bars, Rods and Shapes (B 107-58), 1958 Book of ASTM Standards, Part 2.

[f] Aeronautical Material Specifications.

TABLE II.—YOUNG'S MODULUS OF MAGNESIUM ALLOYS.

Alloy Numbers	Alloy and Temper	Fabrication Process[a] and Form, in.	Initial Grain Size, 0.001 in.	Young's Modulus, 10^6 psi								
				78 F	200 F	300 F	400 F	500 F	600 F	700 F	750 F	800 F
				ELEVATED-TEMPERATURE ALLOYS								
1.......	HM21A-T8	R 0.064 thick	...	6.50	...	6.28	6.08	5.85	5.70	4.83^{9c}	...	2.91^{5c}
2.......	HK31A-H24	R 0.50 thick	...	6.57^{5b}	6.18^{9}	6.15	5.70	4.73	3.70	...	...	...
3.......	HK31A-H24	R 0.064 thick	...	6.42^{9}	6.13	6.07	5.79	5.25	...	...	...	...
4.......	HK31A-O	R 0.50 thick	...	6.51^{11}	6.20^{6}	6.11^{6}	5.69	5.01	3.80	2.61	...	...
5.......	HK31A-O	R 0.104 thick	...	6.45^{8}	6.25	6.13	5.93	5.40	4.30^{2}	...	2.43	...
6.......	HK31A-T6	R 0.50 thick	6	6.40^{13}	5.97^{6}	5.94	5.81	5.46	5.36	...	...	...
7.......	HM31XA-F	E 0.5 diameter	0.68	6.46^{8}	6.18	6.03	5.82	5.76	5.51	...	...	...
8.......	HM31XA-F	E 0.5 diameter	0.13	6.60	6.32	6.10	5.98	5.73	5.45	...	...	...
9.......	HM21A-T5	F Frame	...	6.66	...	...	6.01	...	5.61	4.2^{1}	...	...
10.......	HZ32A-T5	C 0.75 × 12 × 14	2	6.55^{24}	6.33	5.94	6.18	5.72	5.85^{6}	...	...	...
11.......	HK31A-T6	C 0.75 × 12 × 14	3	6.49^{11}	6.26	6.08	5.81^{6}	5.62	5.56^{6}	...	...	...
12.......	EK30A-T6	C 0.75 × 12 × 14	5	6.49	...	6.19	...	5.93	5.45	...	...	...
13.......	EK41A-T6	C 0.75 × 12 × 14	...	6.47	...	6.21	...	5.84	5.56	...	...	...
14.......	EZ33A-T5	C 0.75 × 12 × 14	2	6.46	...	5.96	6.06	5.59	5.42	...	...	...
15.......	ZH62A-T5	C 0.75 × 12 × 14	...	6.73^{10}	6.40	6.15	5.85	...	5.32^{6}	...	...	...
16.......	HM11XA-F	DC Part (0.150 thick)	0.8	6.41^{6}	...	...	5.79	...	5.58^{6}	5.45	...	5.02
				ROOM-TEMPERATURE ALLOYS								
17.......	AZ31A-H24	R 0.50 thick	...	6.34^{8}	6.08	5.17	4.33	3.42^{5}	2.56^{6}	...	...	...
18.......	AZ31A-O	R 0.50 thick	0.9	6.42^{12}	6.14	5.91	5.00	4.18	3.28^{30}	...	...	...
19.......	AZ81A-T4	C 0.75 × 12 × 14	10	6.34	...	...	5.27	4.52	4.10	...	...	...
20.......	AZ91C-T6	C 0.75 × 12 × 14	6.0	6.51^{7}	6.02	5.72	5.07	4.07	3.53^{6}	...	...	...
21.......	AZ92A-T6	C 0.75 × 12 × 14	12	6.27^{6}	6.03	5.56	4.48	3.63	2.96	...	...	...
22.......	K1X1-F	C 0.75 × 12 × 14	...	5.55^{6}	...	...	...	...	...	...	...	...
23.......	K1X1-F	DC Part (0.150 thick)	...	5.24^{9}	...	...	...	...	...	...	...	...
24.......	(P) ZK60B-F	E 1.5 × 5.25	...	6.51^{6}	...	...	...	...	...	...	...	...
25.......	ZK60A-F	F 0.5 diameter	...	6.56^{2}	...	...	...	...	...	...	...	...

EXPERIMENTAL COMPOSITIONS

26	Sublimed magnesium-O	E	0.5 diameter	1.7	5.77^8	5.18^6	4.82^6	4.90^6	4.85^6	4.36^6	...	...	...
27	Commercial electrolytic Mg-F	E	0.5 diameter	0.8	6.24	5.33	4.65	3.87	3.21	2.40	...	...	...
28	AZ31-T2	C	0.75 × 12 × 14	12.0	6.49^5	6.31	6.03	5.94	5.29	4.44	...	...	...
29	E2-T6	E	0.5 diameter	2.4	6.37^9	6.60	5.99^6	5.84^6	5.56^6	5.38^6	...	...	...
30	E4-T6	E	0.5 diameter	5.0	6.58^5	6.62	6.34	6.03	5.48	4.84	...	...	...
31	EM40-T6	E	0.5 × 3	1.1	6.64^{19}	6.57	6.50	6.10^6	6.06	5.55^6	...	...	...
32	ZE10-H24	R	0.5 thick	0.5^d	6.48^{21}	5.95	5.83^6	4.60^6	3.33^9	2.51^9	1.69	...	...
33	ZE10-O	R	0.5 thick	1.9^d	...	...	...	...	4.66	3.75	2.84	...	...

[a] R = rolled, E = extruded, C = sand cast, DC = die cast, F = forged.

[b] Superscripts indicate number of tests if other than triplicate.

[c] Rapidly heated and tested.

[d] Recrystallized grain size after 600 F test.

tions of thorium or rare earths increase the elevated-temperature relaxed modulus from 4.4×10^6 to 5.4×10^6 psi, while combined aluminum and zinc additions decrease the modulus from 4.4×10^6 to values as low as 3.0×10^6 psi. Figure 1 graphically compares the moduli of the elevated-temperature and room-temperature alloys as a function of temperature.

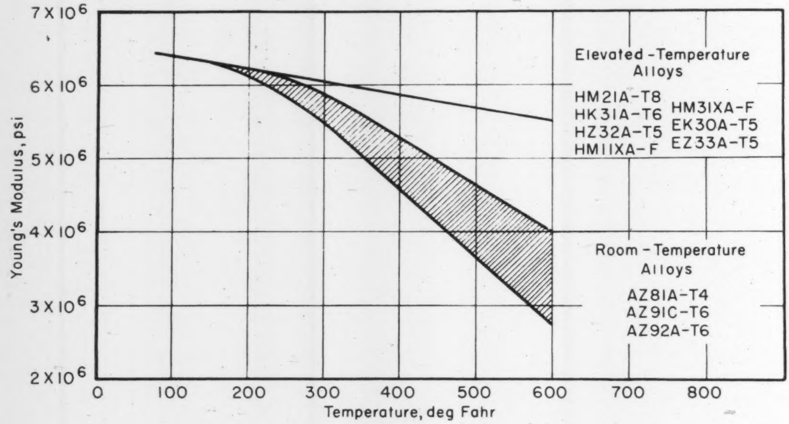

FIG. 1.—Effect of Alloy Content on Young's Modulus of Magnesium as a Function of Temperature.

The relatively high 600 F moduli (5.4×10^6 psi) of the magnesium-rare earth alloys (EK30A-T6, EK41A-T6, EZ33A-T5, E2-T6, EM40-T6) are believed to be the result of Mg_9Ce precipitate keying the grain boundaries against movement as demonstrated by Roberts (7) thus preventing significant anelastic relaxation. Increasing the cerium content in pure magnesium from 2 to 4 per cent increased the size of the precipitate particles as revealed metallographically and decreased the 600 F modulus about 0.5×10^6 psi (see alloys Nos. 29 and 30 in Table II). The larger precipitate particles in the 4 per cent cerium alloy are thought to be less effective in keying the grain boundaries against anelastic relaxation, thus accounting for the decrease in modulus.

The magnesium-thorium alloys (HM21A-T8[6], HK31A-T6, HM31XA-F, HM21A-T5, HZ32A-T5, HM11XA-F) also have relatively high 600 F moduli ($\sim 5.4 \times 10^6$ psi, see Table II) which are thought to result from grain-boundary keying by a magnesium-thorium compound in a manner similar to Mg_9Ce keying in magnesium-cerium alloys. A typical grain-boundary precipitate for a magnesium-thorium-manganese alloy (HM21A-T8) is illustrated in Fig. 2(*a*). The 600 F moduli of the commercial magnesium-thorium and magnesium-rare earth alloys ($\sim 5.4 \times 10^6$ psi) agree well with the value of 5.2×10^6 psi for high-purity magnesium determined ultrasonically by Fredrick (8), and the value of 5.4×10^6 psi for high-purity magnesium determined by Levinson and Graff (9) using a resonant frequency method. The agreement among these data suggests that these elevated-temperature alloys exhibit essentially no anelastic relaxation even when tested in simple tension at 600 F.

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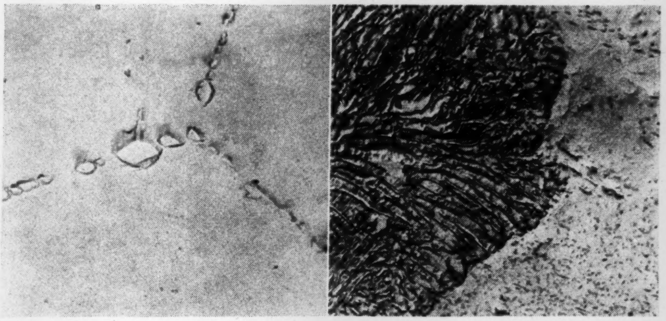
[6] The data shown in Table I for HM21A-T8 sheet, as well as HM21A-T5 forged magnesium, were obtained under the following Wright Air Development Center contracts: (1) AF 33(616)-2337, (2) AF 33(616)-3578, respectively.

It is hypothesized that the relatively low 600 F modulus values for magnesium-aluminum-zinc alloys such as AZ91C-T6 and AZ92A-T6 may result from (1) solid solution effects of aluminum and zinc on the magnesium, and (2) increased anelastic relaxation on the boundaries of the pearlitic $Mg_{17}Al_{12}$ precipitate (see Fig. 2(*b*)). Roberts (**10**) has shown that this discontinuous precipitate increases the number of grain boundaries approximately threefold and that these boundaries as well as original boundaries are significant sources of deformation in creep of a magnesium-10 per cent aluminum alloy. Therefore, it is reasonable to assume that these precipitate-formed grain boundaries would also lead to increased anelastic relaxation as shown for grain boundaries in pure aluminum by Kê (**2,3**).



(*a*) (×16,000), showing magnesium-thorium precipitate at grain boundary of HM21A-T8 magnesium alloy.

(*b*) (×5200), showing pearlitic $Mg_{17}Al_{12}$ precipitate in AZ92A-T6 magnesium alloy (methyliodide etchant).

FIG. 2.—Electron Micrograph.

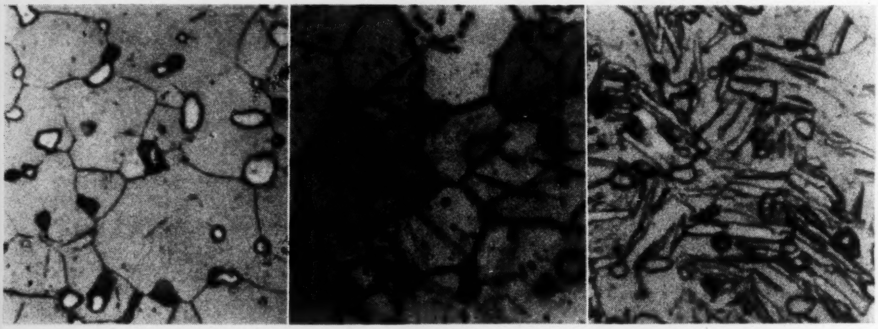
EFFECT OF GRAIN SIZE

Comparisons between the 600 F moduli (see Table II) of rolled HK31A-T6 (alloy No. 6) and cast HK31A-T6 (alloy No. 11), having grain size of 0.006 and 0.003 in. respectively, show no effect of grain size. A similar comparison on another elevated-temperature alloy HM31XA-F (alloys Nos. 7 and 8, having grain size of 0.00068 and 0.00014 in.) also shows no effect as would be expected if the grain-boundary keying hypothesis is correct. It is also interesting to note that although there is a very large difference in grain size between the magnesium-thorium-zirconium and magnesium-thorium-manganese alloys, no significant difference in their 600 F moduli is observed.

A solid-solution alloy containing magnesium-0.8 per cent zinc-0.2 per cent mischmetal (ZE10, alloy Nos. 32 and 33) illustrates the relatively large change in 600 F moduli which can occur upon changing grain size in an alloy which is not designed for elevated-temperature service. Table II shows that increasing the recrystallized grain size from 0.0005 to 0.0019 in. (by annealing) increased the modulus at 500, 600, and 700 F by approximately 1.25×10^6 psi. This grain size effect is consistent with the findings of Kê (**2,3**).

EFFECT OF COLD WORK

An extruded alloy (EM40-T6) containing 4 per cent mischmetal and 0.2 per cent manganese in the solution-heat-treated and aged condition was selected to demonstrate the effects of cold work because it was readily available and demonstrated a relatively high modulus at 600 F (namely 5.5 × 10^6 psi). Prestraining 2 per cent by tension, 2 per cent by compression, and 16.5 per cent by cold rolling reduced the 600 F modulus to 5.0 × 10^6, 3.5 × 10^6, and 3.7 × 10^6 psi respectively. The decreases in the 600 F modulus correlate with the degree of mechanical twinning as seen in the photomicrographs shown in Fig. 3. That is, the larger the amount of twinning the lower the elevated-temperature modulus. This increase in anelastic relaxation with increasing number of mechanical twins is in agreement with the effect of annealing twins on anelastic behavior of copper-manganese alloys as shown by Siefert and Worrell (4). This is reasonable if one considers the twin boundaries to be simply differences in orientation which behave in an anelastic manner similar to grain boundaries. Although slipbands produced by cold work may also contribute to anelastic effects as pointed out by Zener (5) no study of this effect was made in this investigation.



(*a*) Solution-heat-treated and aged, no twinning, $E = 5.5 \times 10^6$ psi.

(*b*) 2 per cent tension strain, slight twinning, $E = 5.0 \times 10^6$ psi.

(*c*) 2 per cent compression strain, considerable twinning, $E = 3.5 \times 10^6$ psi.

FIG. 3.—EM40 Magnesium Alloy (×500), Acetic Picral Etchant.

Comparison between rolled HK31A-H24 and rolled or cast HK31A-T6 shows the 600 F modulus of the strain-hardened material (-H24) to be approximately 1.7 × 10^6 psi lower than the solution-heat-treated and aged material. Similarly, comparison between rolled AZ31A-H24 and a cast experimental alloy AZ31-T2 in the stabilized condition shows the 600 F modulus of the strain-hardened material to be approximately 2.0 × 10^6 psi lower than that for the cast material (see Fig. 4). The decrease in modulus in the latter case would be expected to be even greater if the grain size of the AZ31-T2 were smaller than the 0.012 in. reported in view of the grain-size effect already described for ZE10 alloy.

EFFECT OF SOLUTION HEAT TREATMENT AND AGING

To demonstrate separately the effects of solution-heat treatment and aging, a specimen of 0.064-in. HK31A sheet was (*a*) solution-heat treated 1 hr at 1050 F (-T4 temper), (*b*) rapidly cooled to room temperature with forced air, (*c*) rapidly heated (10 sec) by a resistance-heating method to the test temperature of 600 F, and (*d*) tested at 600 F for various exposure times of 5 sec to 25 min. After 5 sec at 600 F, the material was still considered to be essentially in the -T4 temper and had a modulus of 4 × 10^6 psi. As testing continued at 600 F (a temperature at which aging can occur) the modulus gradually increased to approximately 5.2 × 10^6 psi after 25 min exposure. This value is in good agreement with the 5.4 × 10^6 psi which is expected for HK31A in the aged condition at 600 F, particularly since the accuracy of the autographic recording system necessarily used for these measurements is somewhat less than that of the main investigation.

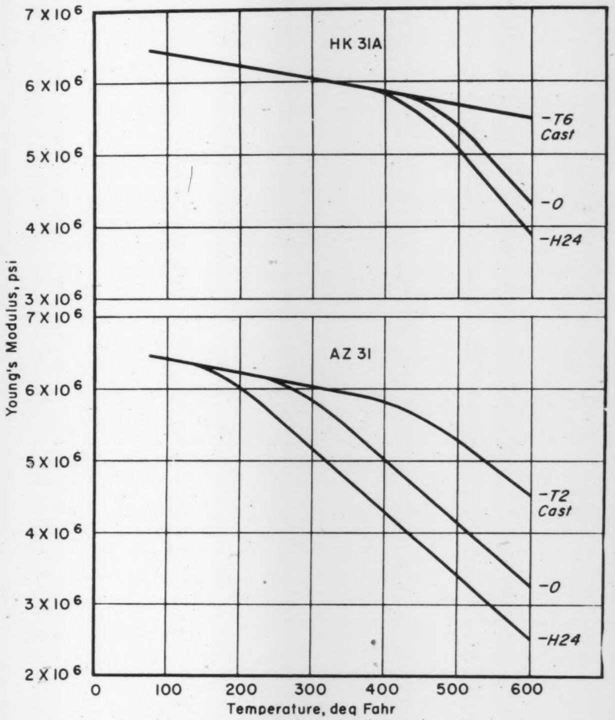


FIG. 4.—Comparison of Cast and Rolled Magnesium Alloys Showing Effect of Strain Hardening and Annealing on Young's Modulus.

Although no similar experiment was performed on alloys in the magnesium-aluminum-zinc system, the work of Roberts (7) and comparison between the 600 F modulus data of the magnesium-aluminum-zinc alloys (Nos. 19, 20, and 21) in this study would lead one to expect solution-heat treatment of these alloys to increase the modulus and aging to decrease the modulus, in direct contrast to the effects noted on HK31A alloy.

EFFECT OF ANNEALING

To show the effect of annealing on an elevated-temperature alloy, HM21A alloy was rolled to the full-hard condition, and specimens were annealed for 1 hr at temperatures of 600 to 900 F and tested at 600 F. The 600 F modulus increased with annealing temperature from 3.5×10^6 psi to 5.5×10^6 psi as shown in Fig. 5. The increase in modulus in this case is attributed to aging and removal of deformation markings (for example, slipbands, twin markings). In a nonaging alloy, such as ZE10 (see alloys Nos. 32 and 33, Table II) annealing may affect the 600 F modulus through (1) removal of certain deformation effects, and (2) changes in grain size resulting from recrystallization and grain growth. Production annealing of commercial material generally results in increased 600 F modulus as can be seen by comparing the -0 and -H24 tempers of HK31A and AZ31A in Fig. 4.

EFFECT OF PROCESSING

Process history (for example, casting, extrusion, rolling) can produce differences in (*a*) the degree of strain hardening, (*b*) grain size, and (*c*) amount and distribution of precipitate, all of which may have significant effects on the relaxed elevated-temperature modulus as already discussed.

The degree of modulus anisotropy in HM21A-T8 sheet alloy was evaluated by comparing several 600 F longitudinal and transverse test results. The average longitudinal value (5.65×10^6 psi, six tests) is within 0.09×10^6 psi of the average transverse value (5.56×10^6 psi, six tests) when tested at 600 F, indicating that the modulus of this material is essentially an isotropic property. This result would be expected in view of the maximum anisotropy of 15 per cent reported for pure magnesium single crystals by Schmid and Boas (11).

Since changes in grain size and orientation have little effect on the elevated temperature modulus of magnesium-thorium or magnesium-mischmetal alloys, process control for this property is less critical in the elevated temperature alloys than in the room-temperature alloys.

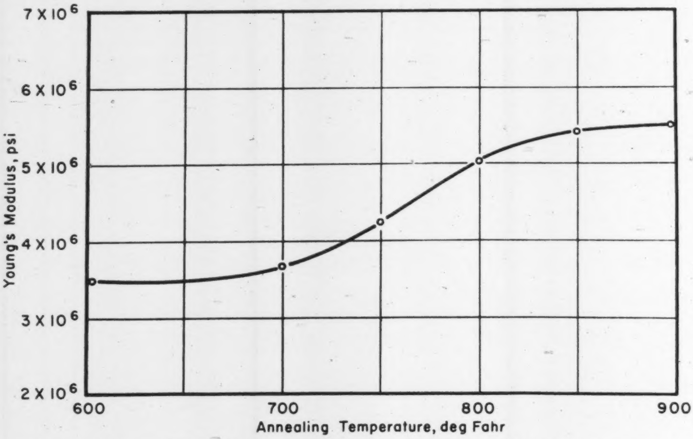


FIG. 5.—Effect of Annealing Temperature (1 hr exposure) on 600 F Young's Modulus of Cold Rolled Magnesium-Thorium-Manganese Sheet (HM21A Alloy).

CONCLUSIONS

Although metallurgical variables have practically no effect on the modulus of magnesium alloys at room temperature, they significantly affect the relaxed Young's modulus at elevated temperatures. Considerable control of the elevated-temperature modulus can be maintained by control of the metallurgical variables and proper selection of alloy content.

Maximum values of relaxed moduli at elevated temperatures can be obtained by: (1) adding thorium or mischmetal to the magnesium, (2) thermally treating material to remove certain effects of strain hardening and to key the grain boundaries against anelastic relaxation by precipitation of second-phase material, and (3) increasing the grain size of the room-temperature-type alloys.

Minimum values of relaxed moduli at elevated temperatures can be obtained by: (1) strain hardening, (2) decreasing the grain size in room-temperature-type alloys, and (3) adding second-phase material which will not key the grain boundaries but which will create new sites for anelastic relaxation.

The relaxed moduli in this paper may be used to establish the initial portion of typical curves of stress *versus* strain, tangent modulus, or secant modulus.

Elevated-temperature values of the relaxed Young's modulus (which include the measurement of anelastic deformation) can be significantly lower than the unrelaxed values determined dynamically.

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DISCUSSION

MESSRS. W. H. GRAFT[1] AND D. W. LEVINSON[1] (*presented in written form*).—A number of the specimens described in this paper were subjected to elastic modulus determinations at Armour Research Foundation employing a dynamic method. Tests were conducted at room temperature and at 600 F as shown in the accompanying Table III. The results of the static determinations conducted by the author of the paper are listed for comparison. It may be noted that the values of the room temperature elastic moduli as measured by the two methods show good agreement but that marked differences were found in the 600 F measurements. This confirms the author's general argument concerning the effect of time-dependent strain as constituting a factor in the determination of a relaxed Young's modulus. One exception to this general trend was exhibited by the alloy HK31A-T6 in which comparable values for the dynamic and static modulus were found. This implies that, although tested under conditions favoring relaxation, an unrelaxed value was obtained.

The degree of anelastic relaxation exhibited by an alloy designed for high-temperature service (HK31A-H24) is less than that of a room temperature type alloy (AZ31A-H24) as shown by a comparison of the respective differences in dynamic and static modulus values determined at 600 F. The former showed a decrease of 1.84×10^6 psi and the latter 2.89×10^6 psi.

An even more graphic example concerns the comparison of the static and dynamic modulus values of HK31A-T6 at 600 F with any of the others reported. In this case the static and dynamic moduli are within 3 per cent of one another.

Very considerable experience with the dynamic method which has been adequately described elsewhere[2, 3] has indicated that the values are reproducible to within ±1 per cent of the modulus reported. This points up, then, the fact that the value of elastic modulus which one obtains from a tension test (especially at elevated temperatures or at room temperature for materials affected by anelastic relaxation) would be expected to be strain rate dependent. At low values of strain rate the number obtained would be smaller and would approach the unrelaxed value obtained by dynamically testing in the kilocycle frequency range only at very high strain rates.

It is suggested that this difference between static and dynamic modulus might be of use in predicting creep resistance, the more creep-resistant the material at a given temperature, the closer together the static and dynamic moduli. As normally tested, the strain rate differences between these tests are large enough to clearly indicate any tendency to relaxation.

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[1] Armour Research Foundation, Illinois Institute of Technology, Chicago, Ill.

[2] W. H. Graft, D. W. Levinson, and W. Rostoker, "The Influence of Alloying on the Elastic Modulus of Titanium Alloys," *Transactions*, Am. Soc. Metals, Vol. 49, pp. 263–279 (1957).

[3] W. H. Graft and W. Rostoker, "The Measurement of Elastic Modulus of Titanium Alloys," Symposium on Titanium, pp. 130–143 (1956). (Issued as separate publication *ASTM STP No. 204*.)

MR. R. W. FENN, JR. (*author*).—The contribution of Messrs. Graft and Levinson is indeed a valuable one, in that their dynamic modulus data allow a direct comparison to the statically determined modulus data in this paper, thereby permitting the relaxation characteristics of several of the alloys to be compared. The conclusions which can be drawn from this data comparison are worth emphasizing.

At room temperature, both the static and dynamic test methods give approximately the same modulus values for the more highly alloyed alloys in both the low-temperature and elevated-temperature category. This indicates that the statically determined moduli for these alloys are essentially unrelaxed. However, the unalloyed magnesium (sublimed magnesium and commercial electrolytic magnesium) shows lower static moduli than dynamic moduli indicating that significant relaxation is occurring in the static evaluation of pure magnesium at room temperature. Since both sublimed magnesium and K1X1 alloy show a relatively low elastic modulus as evaluated by the static tension method at 75 F, it would be intersting to see if the K1X1 alloy exhibits a relatively low elastic modulus by the dynamic test method at 75 F as does sublimed magnesium.

At 600 F, all of the static moduli in the comparison are considerably lower than the dynamic moduli except for the HK31A-T6 alloy. This points up (1) the magnitude of the relaxation effects at elevated temperatures and the importance of selecting the proper test method for determining Young's modulus of magnesium alloys for any particular elevated-temperature application, and (2), the marked effect which cold work has on the relaxation characteristics of magnesium alloys.

TABLE III.—COMPARISON OF DYNAMIC AND STATIC ELASTIC MODULI FOR MAGNESIUM ALLOYS.

Specimen	Alloy and Temper	Modulus at Room Temperature		Modulus, 600 F	
		Static	Dynamic	Static	Dynamic
No. 2	HK31A-H24	6.57×10^6	6.46×10^6	3.70×10^6	5.54×10^6
No. 6	HK31A-T6	6.40	6.31	5.36	5.52
No. 17	AZ31A-H24	6.34	6.45	2.56	5.45
No. 26	Sublimed Mg-O	5.77	6.16	4.36	5.41
No. 27	Commercial Electrolytic Mg-F	6.24	6.53	2.40	5.79
No. 28	AZ31A-T2	6.49	6.41	4.44	5.47

MR. G. R. GOHN.[4]—This paper is important not only because it gives us more precise information that can be used for engineering design but because it points up the necessity of changes in our current educational practices which now lead a good many designers astray.

Most courses in mechanics and materials of design teach the prospective engineer that the elastic modulus for a given material is constant and is not substantially affected by variations in processing or minor variations in composition. As a result, many designs are made on the basis that this assumption is true.

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From the data submitted by the author it is obvious that this assumption of a constant value for the elastic modulus is not true and one can get into very serious difficulties if designs are based on this assumption. For example, you might have insufficient cross-section to give stability to the design.

Furthermore, I think this paper is highly important because it points up the "fuzzy" thinking of many of our theoreticians. During the past two days I sat through a whole series of lectures in which very modern techniques were used to explain theories, particularly theories on the mechanism of fatigue. Dislocation measurements, measurements of slip, etc., were all used to explain the observed phenomena and then the authors turned to an analysis using stress as the criterion for measuring things that were happening in the plastic range. In these analyses the authors obtained stress values merely by multiplying Young's modulus by strain. Obviously when things like this are done there can be no sound design. So it seems to me that papers such as the one which the author has given will not only provide more accurate information for design but will give us suggestions for changing our thinking as to the analysis of phenomena in terms of stress and strain.

MR. H. G. WARRINGTON.[5]—I heartily endorse the statement of Mr. Gohn, in that this work is most important in throwing more light on the mystery of what is modulus of elasticity.

The question that occurs to me in looking at the curves shown is whether the author had considered that the thorium type alloys do produce a eutectic of a relatively high melting point and presumably relatively high strength at elevated temperatures. The AZ31 type alloy is a straight solid solution at these temperatures and AZ92 type alloy has a second phase which is a pseudo-eutectic of extremely low melting point and presumably extremely low strength. This of course prompts the suggestion that in the AZ92 type alloy there might be less reproducibility in modulus results and leads one to wonder what would happen if one adds zinc to the thorium type alloy until a zinc phase appears and what its effect would be.

MR. FENN.—In answer to Mr. Warrington's first question, the differences in the moduli observed in the magnesium-thorium and magnesium-aluminum-zinc systems were considered primarily from the standpoint of the properties of the component phases and how these phases might interact to affect the over-all elastic behavior. Although one might expect increases in Young's modulus in accordance with the law of mixtures upon the addition of compounds having higher moduli (based on the work of Köster et al,[6] it is the opinion of the author that the dispersion, size, and location of the precipitated compound is a more important factor than the modulus or strength (within limits) of the compound in determining the relaxation characteristics and therefore the static elastic moduli of magnesium alloys at elevated temperatures. With respect to the second question concerning the effect of zinc additions on the thorium type alloys, comparison between the moduli of HZ32A-T5 (containing 2 per cent zinc) and HK31A-T6 (alloys Nos. 10 and 11, respectively in Table II) shows the HZ32A-T5 to have slightly higher modulus at 600 F. However, the difference between the moduli of the two alloys could possibly be accounted for on the basis of experimental errors. For this reason the two alloys are considered to have equivalent moduli up to 600 F as shown in Fig. 1.

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[6] W. Köster und W. Rauscher, "Beziehungen zwischen dem Elastizitätsmodul von Zweistofflegierungen und ihrem Aufbau," *Zeitung für Metallkunde*, Vol. 39, pp. 111–120 (1948).

PREDICTION OF LONG-TIME CREEP WITH TEN-YEAR CREEP DATA ON FOUR PLASTIC LAMINATES*

By W. N. Findley[1] and D. B. Peterson[1]

Synopsis

Long-time creep data for tension creep tests of four plastic laminates are reported for testing times up to about ten years' duration at constant load and constant temperature of 77 F and relative humidity of 50 per cent: a grade C canvas laminate, two paper laminates, and an asbestos laminate. Each of these sets of data has been compared with the equation $\epsilon = \epsilon_0 + mt^n$ and the stress dependence of ϵ_0 and m have been evaluated in terms of hyperbolic sine functions. Good agreement was obtained in most tests.

The data for the first 2000 hr of test of the grade C canvas laminate and the parallel-laminated paper laminate were reported in a paper published in 1944, together with constants for the above-mentioned equation. These constants were used to predict the creep for the remainder of the creep tests, the duration of the tests being on the order of 90,000 hr. Surprisingly good agreement with test data was obtained over that interval. Predictions for the same time intervals investigated from four other equations used to describe creep were unsatisfactory.

During 1943–1944, a series of creep tests of plastic laminates was started for tests at several constant loads in a room maintained at a constant temperature of 77 F and a relative humidity of 50 per cent. The materials included a grade C canvas laminate, a parallel-laminated paper laminate, a cross-laminated paper laminate, and an asbestos laminate. In 1944 the results of the first 2000 hr, approximately 83 days, were reported for the grade C canvas laminate and the parallel-laminated paper laminate (**1**).[2] It was shown in the 1944 paper (**1**) that there was a very satisfactory agreement between the data and the equation

$$\epsilon = \epsilon_0 + mt^n \qquad (1)$$

where ϵ is the strain, t is time, n is a constant which was found to be independent of stress, and ϵ_0 and m are constants for a given stress which were since found to be describable as hyperbolic sine functions of stress for the canvas laminate (**2**). The value of ϵ_0 was found to equal zero for the parallel-laminated paper laminate. The tests of the grade C canvas laminate were since continued to approximately 85,000 hr, more than 40 times the duration of the first reported tests; and the tests of parallel-laminated paper laminate were continued to about 92,000 hr.

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* Presented at the Sixty-first Annual Meeting of the Society, June 22–27, 1958.

[1] Professor of Engineering and Student, respectively, Division of Engineering, Brown University, Providence, R. I.

[2] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 855.

In the present paper, the data for the remaining long-time portion of these tests are presented, together with the prediction based on the constants in Eq 1 which were determined from the data for the first 2000 hr and published in 1944. Also presented are long-time creep data on the two other laminates listed above, together with an analysis of these data including comparisons with Eq 1 and the hyperbolic sine stress functions. Data for recovery after removal of load are included for all materials together with creep after reloading for two of the materials.

Several other types of equations have been proposed by other investigators in the literature. Among these are the following:

Leaderman (3) expressed creep of Bakelite under torsion as:

$$\epsilon = \epsilon_0 + A \log_e t + Bt \ldots\ldots\ldots (2)$$

where ϵ is the strain, t is the time, and ϵ_0, A, and B are constants which are functions of stress and of the material.

The same type of equation has been used by Telfair, Carswell and Nason (4) for phenolic plastics and later by Lyons (5) for tire cords.

Cottrell and Aytekin (6) used a modification of the Andrade equation for single crystals of metals:

$$\epsilon = \epsilon_0 + At^{\frac{1}{3}} + Bt \ldots\ldots\ldots\ldots (3)$$

where ϵ is the strain at time t, ϵ_0 is the instantaneous strain, and A and B are constants.

The following expression for creep derived from the motion of a Kelvin and a Maxwell unit in series (7) has been used frequently,

$$\epsilon = \epsilon_0 + A(1 - e^{-ct}) + Bt \ldots\ldots (4)$$

where constants ϵ_0, A, B, and c are functions of stress and of the material.

MATERIALS

Grade C Canvas Laminate:

The composition and processing of this laminate were fully described in reference (8). It was molded at a pressure of 1800 psi with a cresol and formaldehyde resin. The grade C canvas laminate employed in these tests was the same from which test specimens were obtained for other tests previously reported (1,2,8–11). The creep tests reported for this laminate in the present paper are the same tests reported for this material in references (1) and (8) and are the same as reported in reference (9) as having been started in August 1943.

Asbestos Laminate:

This laminate was supplied by the Synthane Corp. in 1943. It was a grade AA laminate made of 18 oz per sq yd asbestos fabric having a thread count of 18 × 16. The resin employed was Bakelite Corp.'s No. 2427, composed of xylenol plus formaldehyde with a small amount of cresol and a solvent of ethyl alcohol. A single stage process was used. The laminate was formed of 20 plies of fabric, parallel laminated, containing 47 per cent resin. The press was heated with hot water to a temperature of 340 F. The laminate was pressed at 1800 psi and 340 F for 50 min, then cooled for 20 min, at which time the temperature was 100 F.

Parallel-Laminated Paper Laminate:

The composition and processing of this laminate were fully described in reference (12). The laminate was molded of Mitscherlich paper with a phenolic resin using a low molding pressure (230 psi). The creep tests reported in the present paper for this laminate are the same tests reported for shorter times in references (1) and (12).

Cross-Laminated Paper Laminate:

The composition and processing of this laminate were fully described in reference (8). It was molded of Mitscherlich paper with a phenolic resin using a low pressure of 250 psi. The creep tests reported in the present paper for this laminate are the same tests reported for shorter times in reference (8).

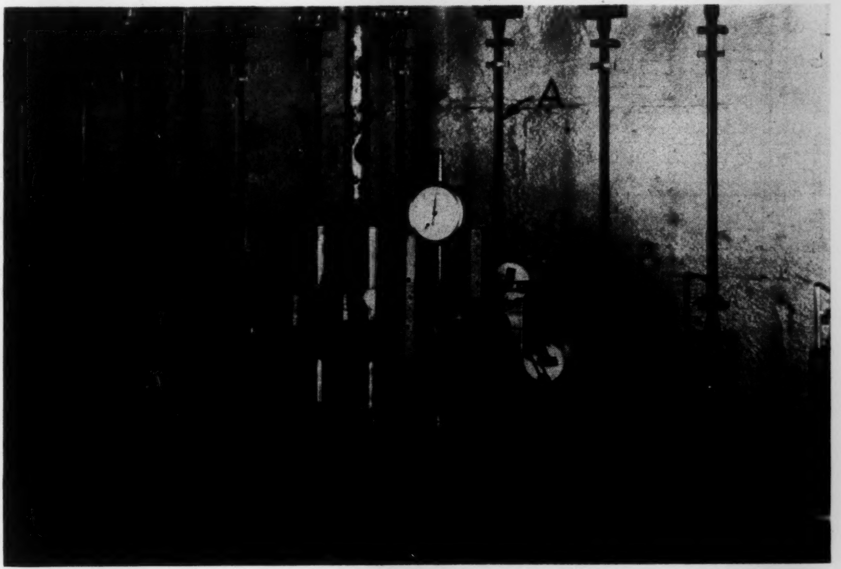


FIG. 1.—Apparatus for Tension Creep Testing.

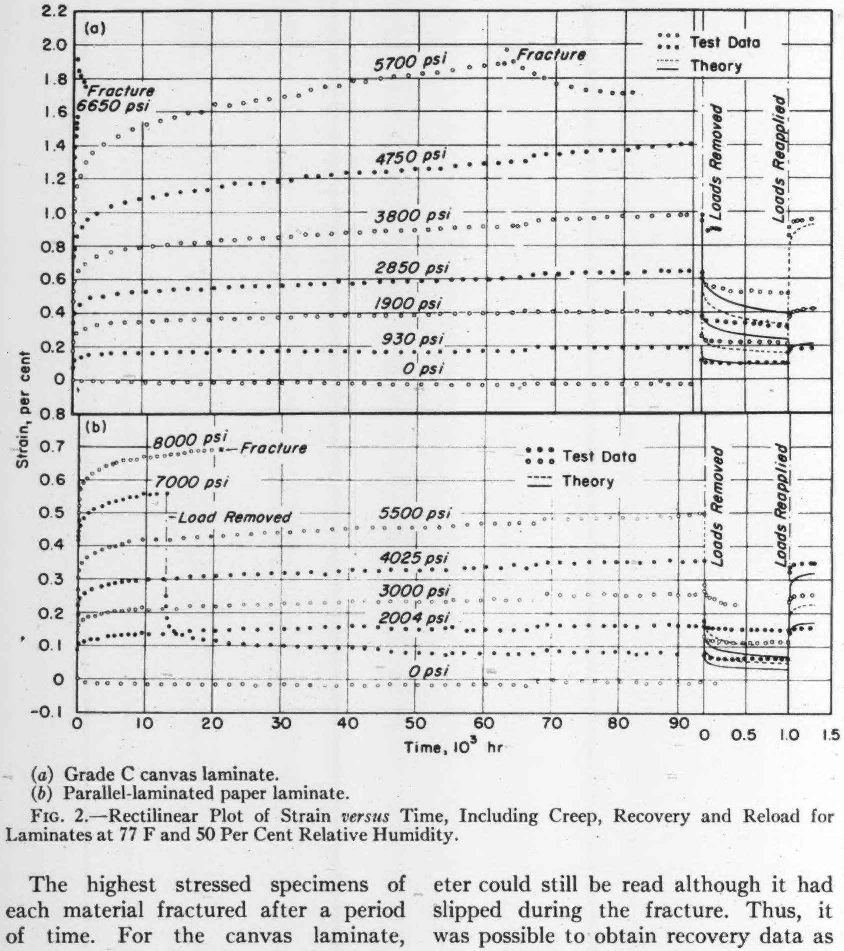
APPARATUS, SPECIMENS AND TEST PROCEDURE

The test specimens and procedure employed for the creep tests were the same as previously described (8). The apparatus, shown in Fig. 1, consisted of a frame and levers for applying tension loads to the specimens, *A*, and lever type extensometers, *B*, read by means of a measuring microscope, *C*, to determine the strains.

TEST RESULTS

In Fig. 2 are shown results of long-time creep tests of grade C canvas laminate, parallel-laminated and cross-laminated paper laminates, and asbestos laminate, respectively, for tests at several stresses. In addition to the creep data, Fig. 2 contains recovery data and Figs. 2(*a*) and (*b*) contain creep data obtained from reloading following a period of recovery at no load.

The creep behavior of all the materials was similar in that the rate of creep was rapid at first but diminished continuously with time. From the strain resulting from a given stress in the long-time creep data, Fig. 2, it was found that the canvas laminate and the asbestos laminate were least resistant to creep and the cross-laminated paper laminate was most resistant to creep. A further consideration of this comparison is presented in a later section.



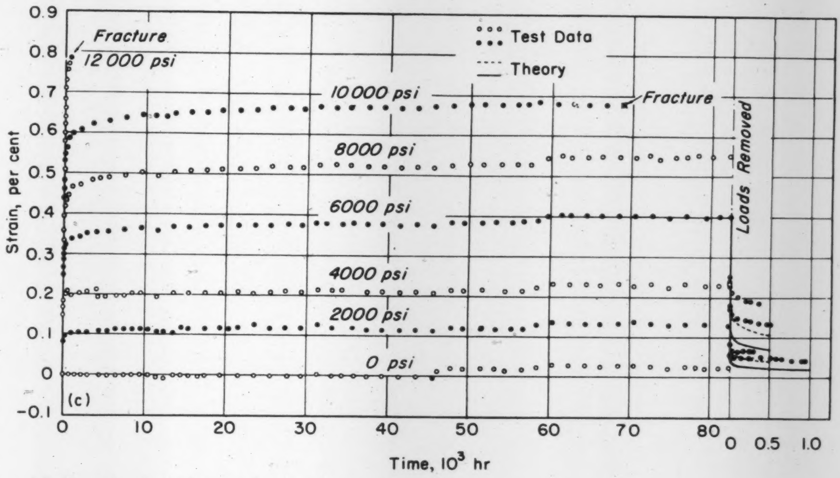
(*a*) Grade C canvas laminate.
(*b*) Parallel-laminated paper laminate.

FIG. 2.—Rectilinear Plot of Strain *versus* Time, Including Creep, Recovery and Reload for Laminates at 77 F and 50 Per Cent Relative Humidity.

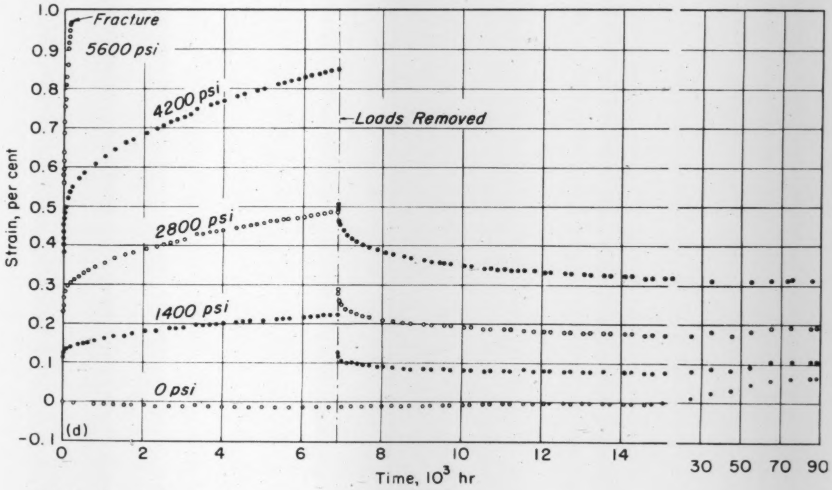
The highest stressed specimens of each material fractured after a period of time. For the canvas laminate, the parallel-laminated and the cross-laminated paper laminates, a specimen fractured after a very long time, 62,000, 22,000, and 69,000 hr, respectively. It is very unusual to obtain creep-rupture data for such long times as these for any material—metals or plastics.

Two specimens of grade C canvas laminate fractured outside the gage length in such a way that the extensometer could still be read although it had slipped during the fracture. Thus, it was possible to obtain recovery data as shown in Fig. 2(*a*) for the tests at 6650 psi and 5700 psi. The strains indicated are, of course, much too large because of slippage of the extensometer, but the change in strain after removal of the load is correctly indicated.

The specimen of parallel-laminated paper laminate tested at 7000 psi was unloaded at 13,000 hr, and recovery readings were made for about 80,000 hr



(*c*) Cross-laminated paper laminate.

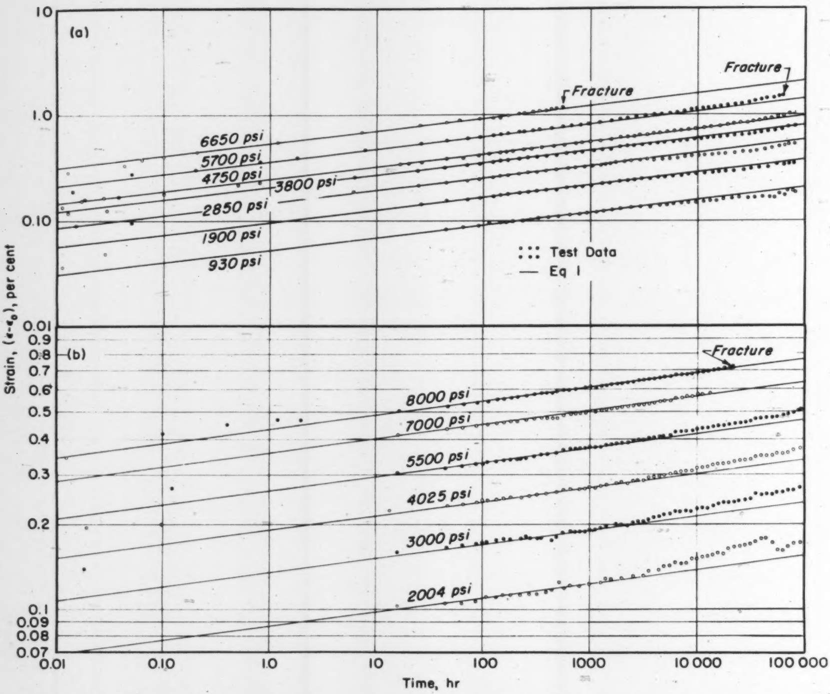


(*d*) Asbestos laminate.

FIG. 2.—*Continued.*

as shown in Fig. 2(*a*). It was observed that even in this long recovery time the creep was not fully recovered. Approximately 13 per cent of the elongation existing just before unloading was not recovered.

A similar tendency for the recovery to be incomplete was observed in the



(*a*) Grade C canvas laminate (strains corrected for shrinkage).
(*b*) Parallel-laminated paper laminate (strains corrected for shrinkage).

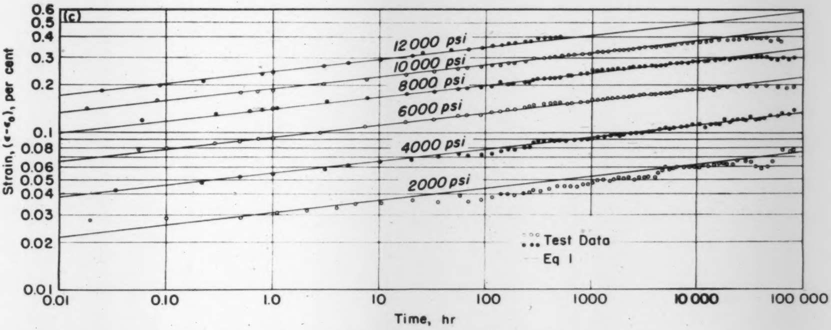
FIG. 3.—Log-Log Plot of Strain *versus* Time for Tension Creep Tests of Laminates at 77 F and 50 Per Cent Relative Humidity.

recovery of the asbestos laminate in which about 80,000 hr of recovery data were obtained following 6900 hr of creep as shown in Fig. 2(*d*). The same tendency was also observed in recovery following long-time tests as shown in Figs. 2(*a*), (*b*), and (*c*).

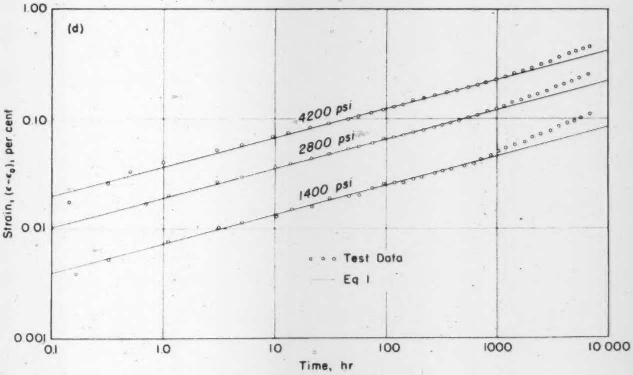
Upon reloading some of the specimens of grade C canvas laminate and parallel-laminated paper laminate, creep occurred again as shown in Figs. 2(*a*) and (*b*). The rate of creep on reloading was very rapid immediately after loading but decreased much more abruptly than on first loading. It appeared that after a short interval the creep deformation continued as though there had been no interruption of the original test stress.

Long-time Prediction from Creep Equation:

In a previous paper (1) the data for the first 2000 hr of creep in the grade C canvas laminate and the parallel-laminated paper laminate were analyzed and found to be described quite well by equations of the form of Eq 1. The values of the constants as determined from the 2000 hr of testing are given in Table I, items *A* and *D*, for the canvas



(*c*) Cross-laminated paper laminate (strains corrected for shrinkage).



(*d*) Asbestos laminate (strains not corrected for shrinkage).

FIG. 3.—*Continued.*

laminate and parallel-laminated paper laminate, respectively. The constants were determined by plotting log $(\epsilon - \epsilon_0)$ *versus* log t and adjusting ϵ_0 until the best straight line was obtained. In reference (1) it was shown that very satisfactory straight lines resulted, which indicated excellent agreement with Eq 1. It was also observed that the lines for tests at several stresses of each material were parallel, indicating that the exponent n was independent of stress.

In the present paper these log-log diagrams have been extended by adding the data for the remaining 87,000 and 89,000 hr, respectively, as shown in Figs. 3(*a*) and (*b*). Also shown in Figs. 3(*a*) and (*b*) are the straight lines representing Eq 1 using the constants determined from the first 2000 hr.[3] These lines thus represent the prediction provided by Eq 1 based on observations for 2000 hr. For the data shown, this is an extrapolation of over fortyfold, which is a prediction of about 10 yr into the future from observations of less

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[3] In Fig. 3(*b*) the straight line shown for the 5500 psi stress has been corrected for the slight error in slope of the line as shown in Reference (**1**). A corresponding correction of ϵ_0 has been made in Table I.

TABLE I.—VALUES OF CONSTANTS FOR EQ 1.

Material	Item	Date Tests Started	Duration of Test Considered in Analysis, hr	Stress σ, psi	ϵ_0, in. per in.	m, in. per in.	n, dimensionless
Grade C Canvas Laminate	*A*	Aug. 1943	2 000	930	0.000255	0.000528	0.1183
				1 900	0.000865	0.000968	0.1183
				2 850	0.001309	0.001475	0.1183
				3 800	0.00215	0.00205	0.1183
				4 750	0.00349	0.00247	0.1183
				5 700	0.00400	0.00363	0.1183
				6 650	0.00409	0.00535	0.1183
	B	Aug. 1943	89 000	930	0.000600	0.000274	0.151
				1 900	0.001500	0.000500	0.151
				2 850	0.002100	0.000859	0.151
				3 800	0.003100	0.001240	0.151
				4 750	0.004000	0.001830	0.151
				5 700	0.005300	0.00241	0.151
	C	Nov. 1953	1 550	2 850	0.00235	0.000461	0.1706
				5 700	0.00504	0.001678	0.1706
Parallel Laminated Paper Laminate	*D*	Aug. 1943	2 000	2 004	0	0.000869	0.0500
				3 000	0	0.001352	0.0500
				4 025	0	0.00191	0.0500
				5 500	0	0.00263	0.0500
				7 000	0	0.00358	0.0500
				8 000	0	0.00434	0.0500
	E	Aug. 1943	91 000	2 004	0.000900	0.000060	0.244
				3 000	0.001320	0.000158	0.191
				4 025	0.001830	0.000250	0.177
				5 500	0.002300	0.000505	0.146
				7 000	0.003000	0.000800	0.131
				8 000	0.003200	0.00140	0.106
	F	Aug. 1943	91 000	2 004	0.000450	0.000368	0.114
				3 000	0.000950	0.000435	0.114
				4 025	0.001400	0.000593	0.114
				5 500	0.001900	0.000820	0.114
				7 000	0.002700	0.00105	0.114
				8 000	0.003400	0.00122	0.114
Cross Laminated Paper Laminate	*G*	Aug. 1944	83 000	2 000	0.000550	0.000308	0.0763
				4 000	0.001200	0.000545	0.0763
				6 000	0.001800	0.000930	0.0763
				8 000	0.002200	0.001405	0.0763
				10 000	0.002800	0.00188	0.0763
				12 000	0.003800	0.00246	0.0763
Asbestos Laminate	*H*	Apr. 1944	6 890	1 400	0.001130	0.0000730	0.267
				2 800	0.002300	0.000191	0.267
				4 200	0.003950	0.000356	0.267

than 3 months' duration. Figures 3(*a*) and (*b*) show that the agreement between the actual and predicted values of creep is excellent for some stresses. The greatest deviations of the predicted creep curves from the data were: about 10 and 15 per cent, respectively, for each of three specimens of the grade C canvas laminate (Fig. 3(*a*); and about 10 per cent or less and 20 per cent for the four highest and two lowest stressed tests, respectively, of the parallel-laminated paper laminate (Fig. 3(*b*)). This percentage was determined by taking the difference between the extrapolated value and the actual creep strain, dividing by the predicted strain and multiplying by 100 per cent. Some of the factors which may have influenced the creep tests, and hence the agreement with the predictions of Eq 1, are considered later.

Analysis of Long-Time Creep Data:

An analysis based on the total duration of the creep tests was made for each material by determining the constants by Eq 1 which best represented the entire data. The constants obtained in this analysis are shown in Table I. For the grade C canvas laminate it was found that the results were of the same order of reliability as those obtained earlier from the analysis of the 2000 hr data and its extrapolation.

During analysis of the complete data for the parallel-laminated paper laminate it was found that the curves for different stresses tended to converge slightly at large times. The constants from this analysis are given as item *E*, Table I. However, by ignoring some of the questionable points at the start of the test, a different set of constants having the same *n* was obtained (shown as item *F* in Table I). These constants made Eq 1 in somewhat better agreement with all the data than the lines shown in Fig. 3(*b*), which were based on the data for the first 2000 hr.

The constants for Eq 1 obtained from analyses of the long-time creep data of the cross-laminated paper laminate and asbestos laminate are given in Table I, items *G* and *H*, respectively. The results of these analyses are shown together with the test data from these two laminates in Figs. 3(*c*) and (*d*), respectively. The agreement between Eq 1 and the test data from the cross-laminated paper laminate was very satisfactory, as shown in Fig. 3(*c*), except where accidental shocks disturbed the tests, especially the tests at the lowest two stresses. The data for the stress of 4000 psi in Fig. 3(*c*) have been corrected for the effect of shock at several points by subtracting the change in the strain indication caused by the shock from all succeeding observations. Corrections for shrinkage were not made in Fig. 3(*d*) because of confusion which could not be resolved in the test data of the control specimen.

When long-time tests were started, the possibility of describing creep by Eq 1 had not been considered, so the importance of readings at short time intervals for use in log-log plotting was not recognized. Thus, very few creep data were recorded for times less than 10 hr.

As reported earlier (2,13), it was found that the exponent n in Eq 1 was independent of stress and that the coefficients ϵ_0 and m could be expressed as hyperbolic functions of stress

$$\epsilon_0 = \epsilon_0' \sinh \sigma/\sigma_\epsilon \quad \text{and} \quad m = m' \sinh \sigma/\sigma_m ,$$

where ϵ_0', σ_ϵ, m', and σ_m are constants. Thus Eq 1 could be written as a function of stress as well as time as follows:

$$\epsilon = \epsilon_0' \sinh \sigma/\sigma_\epsilon + m't^n \sinh \sigma/\sigma_m \ldots (5)$$

The constants ϵ_0', σ_ϵ, m', and σ_m have been evaluated for the four materials tested and are given in Table II. The procedure employed in evaluating these constants was described previously (13).

A comparison of creep of the four laminates was made by comparing their constants in Eq 1 as listed in Table I. The values of m and n determine the time-dependent behavior. Creep resistance is the greatest when m and n have the smallest values. Comparing the four laminates for stresses of approximately 4000 psi and using the analyses of the entire data, it was observed that the cross-laminated paper laminate had the smallest value of n with increasingly larger values for the parallel-laminated paper laminate, canvas laminate, and asbestos laminate. This is consistent with the qualitative comparison made above on the basis of total strain. The asbestos laminate had the smallest value of m with increasingly larger values for the cross-laminated paper laminate, parallel-laminated paper laminate, and canvas laminate.

TABLE II.—CONSTANTS FOR CREEP EQUATION, INCLUDING STRESS FUNCTIONS, EQ 5.

Material	Date of Test	Duration of Test Considered in Analysis, hr	n	ϵ_0', in. per in.	m', in. per in.	σ_ϵ , psi	σ_m , psi
Grade C Canvas Laminate	Aug. 1943	2 000	0.1183	0.001875	0.001875	4 000	4 000
	Nov. 1953	1 550	0.1706	0.00609	0.000303	7 560	2 362
Parallel Laminated Paper Laminate	Aug. 1943	91 000	0.114	0.00184	0.002019	6 000	14 000
Cross Laminated Paper Laminate	Aug. 1944	83 000	0.0763	0.00315	0.001176	12 000	8 000
Asbestos Laminate	Apr. 1944	6 890	0.267	0.00311	0.0001271	4 000	2 400

Influencing Factors:

Over a 10-yr period many things can happen which may influence test results. The temperature and relative humidity were maintained constant at 77 ± 1 F and 50 ± 2 per cent, respectively, throughout the testing interval except for a number of times when the air conditioning failed to function properly. These failures were detected by a warning system and were usually corrected within a few minutes to a few hours. In one instance, however, the humidity was not in control for 35 hr. The temperature was rarely affected. However, these conditions undoubtedly had some effect on the creep.

Aging of the material probably had the greatest influence. Aging of the grade C canvas laminate was investigated by performing additional creep tests about 10 yr after the first tests were started, as described in reference (9). The specimens employed in the additional tests had been stored at test conditions since start of the first tests. The amount of creep was reduced by aging. At 500 hr the creep was reduced with age from 1.17 to 0.99 per cent at 5700 psi and from 0.42 to 0.37 per cent at 2850 psi. The constants for Eq 1 obtained from tests before and after aging of the canvas laminate are given in Table I, items *A* and *C* respectively. It was found that ϵ_0 and n increased, while m decreased with age. Thus, after aging ϵ_0 and m were not equal, as was approximately true of the original tests. The aging may have resulted from evaporation of water or other volatile constituents, further polymerization or degradation of the polymer or fabric.

Another factor having some effect on creep was the occurrence of uncontrollable shocks to which the equipment was occasionally subjected. These shocks resulted from fracture of other creep specimens or from fracture of specimens in large testing machines in the same building. The extensometer of the 4000 psi specimen of cross-laminated paper

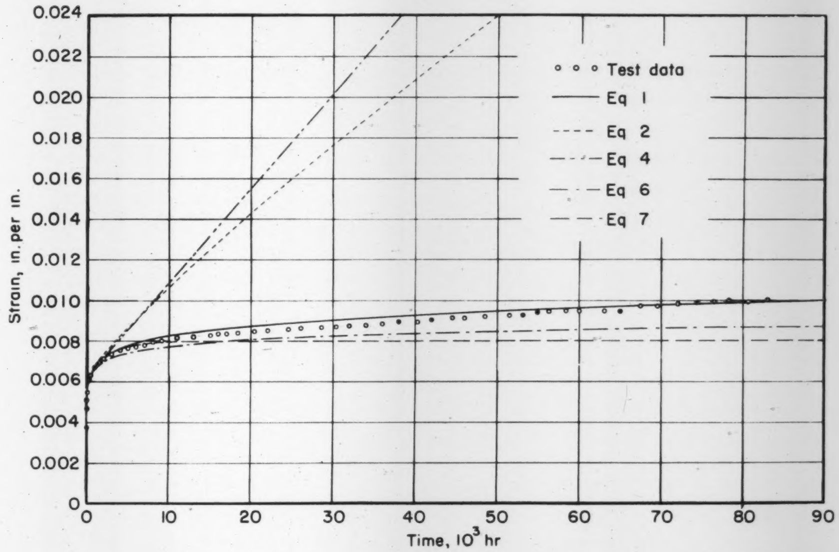

FIG. 4.—Rectilinear Plot of the Creep Test of Grade C Canvas Laminate at 3800 psi, Together with the Prediction of Several Theories Based on the Best Representation for the First 2000 hr.

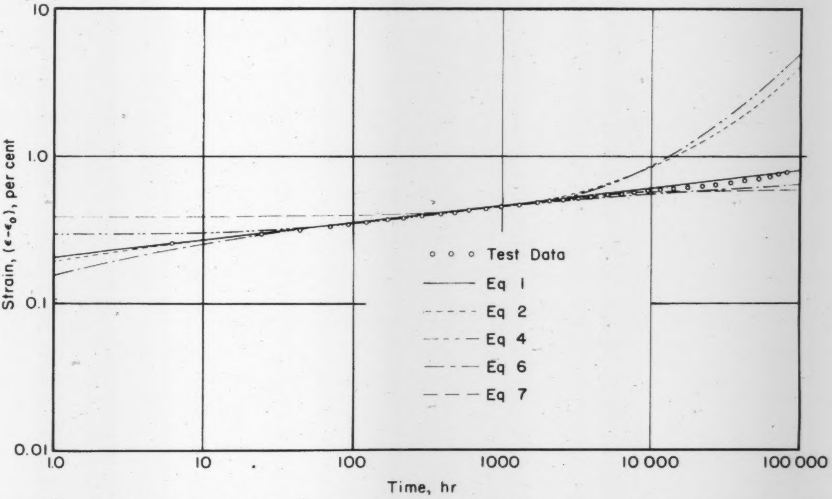

FIG. 5.—Log-Log Plot of the Creep Test of Grade C Canvas Laminate at 3800 psi, Together with the Prediction of Several Theories Based on the Best Representation for the First 2000 hr.

laminate was especially sensitive to shocks, which evidently caused slippage of the extensometer, as may be observed in the data of Fig. 2(*c*).

COMPARISON OF PREDICTION OF CREEP FROM DIFFERENT EQUATIONS

In order to compare the prediction of some of the different equations that have been proposed for describing creep, Eqs 2 and 4 were tried. The constants for these equations which would provide the best representation of the first 2000 hr of test data were determined for the test of grade C canvas laminate at a stress of 3800 psi. This particular test was chosen as representing neither the best nor the poorest agreement with Eq 1, as shown in Fig 3(*a*). The best representations of Eqs 2 and 4 were judged by inspection on the basis of the best agreement between the test data and the equation considering both rectilinear and log-log plots. The resulting equations were then extrapolated to 89,000 hr. The results are shown in Figs. 4 and 5 for rectilinear and log-log plots, respectively. The scales shown for the former were chosen to permit showing the full extent of the test. (Different scales were used in determining the best set of constants.) The values of the constants determined for the different equations are given in Table III.

TABLE III.—VALUES OF CONSTANTS FOR EQUATIONS PLOTTED IN FIGS. 4 AND 5.

Equation Number	ϵ_0, in. per in.	*m*, in. per in.	*n*	*A*, in. per in.	*B*, in. per in.-hr	C, hr^{-1}
Eq 1	0.00215	0.00205	0.1183	...	...	...
Eq 2	0.00407	...	...	0.000769	3.46×10^{-7}	...
Eq 4	0.00512	...	...	0.00110	4.65×10^{-7}	0.00548
Eq 6	0.00375	...	...	0.001	...	...
Eq 7	0.00604	...	...	0.00196	...	0.0004055

It was observed that, whereas the extrapolation of Eq 1 beyond 2000 hr described the test data quite satisfactorily over the entire 89,000 hr of available test data, the extrapolation of Eqs 2 and 4 predicted creep greatly in excess of that actually observed. The largeness of the discrepancies in the predictions of Eqs 2 and 4 was due to the linear term in each equation. So it was decided to investigate the prediction that would be obtained by dropping the linear term from each of Eqs 2 and 4. The resulting equations were, respectively,

$$\epsilon = \epsilon_0 + A \log_e t \qquad (6)$$

$$\epsilon = \epsilon_0 + A(1 - e^{-ct}) \qquad (7)$$

Equation 7 is the form of creep equation predicted by a model consisting of a Kelvin unit in series with an elastic element.

The constants in Eqs 6 and 7 were determined as before to yield the best representation of the data for the first 2000 hr. The extrapolations of the resulting expressions are shown in Figs. 4 and 5. It was observed that both equations predicted too little creep. In fact, Eq 7 was asymptotic to the line $\epsilon = \epsilon_0 + A$ and hence predicted a nearly constant strain after about 10,000 hr. Equation 7 could be made to fit only a very limited range of the data. For the constants given in Table III, Eq 7 described the test data reasonably well from 500 to 2200 hr. It would not fit well for either smaller or larger values of time.

The fact that Eq 6 was in reasonably good agreement with the test data can be shown to be fortuitous. This has been demonstrated by the fact that creep of the poly(vinyl chloride) previously shown to be satisfactorily described by Eq 1 (**13**) could not be described with any degree of satisfaction by Eq 6. This was due to the fact that n of Eq 1 was much larger (0.3109) for the poly(vinyl chloride) than for the canvas laminate.

In view of the tendency of the creep test data of the asbestos laminate toward an upward curvature on the log-log plot, Fig. 3(*d*), the possibility that Eq 4 might describe the data better than Eq 1 was investigated. It was found that Eq 4 could be made to describe the data only over a very limited range. It would not describe either the early or late portion of the test when adjusted to describe the middle portion.

Thus it may be concluded that of the equations considered Eq 1 yields the most accurate prediction for all the materials investigated.

ANALYSIS AND DISCUSSION OF RECOVERY AND RELOAD TESTS

The Superposition Principle:

In a previous paper (**14**) an extension of the tests reported in reference (**13**) was presented. These tests included creep under a sequence of different stresses. The analysis of these tests showed (**14**) that the superposition principle was the most successful of four theories considered for predicting creep resulting from a change in stress.

The superposition principle was applied to the present long-time tests to predict recovery and reload creep. In the tests considered in the present paper, the changes in stress in any test are of the same magnitude on both loading and unloading. Hence, the difficulty due to nonlinearity in the stress term as discussed previously (**14**) did not complicate the present analysis. The superposition principle when applied to recovery following creep requires that the strain at a given time during recovery be equal to the algebraic sum of the following strains: the strain that would have resulted had the creep test continued at constant stress to the given time and the strain that would result at the given time from a stress equal in magnitude but opposite in sense to the original stress applied to an untested specimen at the time corresponding to unloading. In terms of Eq 1 the strain during recovery may thus be written:

$$\epsilon = \epsilon_0 + mt^n - [\epsilon_0 + m(t - t_1)^n],\ t > t_1$$

or

$$\epsilon = mt^n - m(t - t_1)^n \ldots\ldots\ldots (8)$$

where t_1 is the time at which unloading occurs.

Similarly, the strain that would occur following reloading at time t_2 would be given by:

$$\epsilon = \epsilon_0 + mt^n - m(t - t_1)^n + m(t - t_2)^n,\quad t > t_2 > t_1 \ldots (9)$$

Equations 8 and 9 were employed to calculate the recovery and reloading creep respectively for grade C canvas laminate and the parallel-laminated paper laminate. Recovery was also computed for the cross-laminated paper laminate, but was not computed for the asbestos laminate owing to the poor agreement between the creep data and Eq 1 for this laminate. These computations were made using the values of the constants for the creep tests given in Table I, items A, D, and G, respectively. No corrections were made for any lack of agreement between the test data and the predictions of the theory at the time of unloading. The computed recovery and reload creep are shown for comparison with test data of canvas laminate and parallel-laminated paper laminate in Figs. 2(*a*) and (*b*). The comparison for recovery only of cross-laminated paper laminate is shown in Fig. 2(*c*).

The magnitudes of the strains predicted both for recovery and for creep on reloading were in fair agreement with the test data. For the grade C canvas laminate, the predictions and test results were best for the lowest stresses. The predictions were low for recovery from higher stresses. For the paper laminates, the predicted recovery curves were low for all stresses. However, the predicted rate of recovery and predicted rate of creep on reloading were substantially greater than the corresponding rates actually observed. The rate of recovery following creep predicted by the superposition principle for a thermoplastic, poly(vinyl chloride), was also found to be greater than the observed rate of recovery as reported earlier (15). These observations suggest that the superposition principle is not entirely reliable. However, these observations may result, in part, from aging of the materials. This aging may possibly have affected the creep behavior on unloading and reloading.

It should be noted that the extensometers employed were not originally intended to follow reversals of strain, so that some error due to back-lash in the mechanism may have influenced the recovery tests. The fact that the agreement between prediction and observed data on reloading was better than during recovery suggests that some of the extensometers may not have followed reversal of strain accurately.

CONCLUSIONS

In view of the data presented for creep tests at several stresses covering a duration of approximately ten years on several plastic laminates, it was concluded that the equation $\epsilon = \epsilon_0 + mt^n$ is a useful expression for describing creep and for predicting long-time creep of the plastic laminates investigated at constant stress, temperature, and humidity. This equation more closely represented long-time creep tests of the laminates reported than other creep equations investigated.

Acknowledgment:

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The authors are grateful to the following people for assistance in this project: H. E. Anderson, R. G. Carlier, G. E. Chicoine, R. E. Duff, E. Farrell, R. P. Quinlan, and R. A. Wright for taking creep data; F. G. Fernald for computations for Figs. 10 and 11, and E. Szczepanski for preparation of figures.

The gifts of the samples of laminates tested are sincerely appreciated. The grade C canvas laminate and the grade AA asbestos laminate were supplied by the Synthane Corp., Oaks, Pa., the parallel-laminated paper laminate by the Taylor Fiber Co., Norristown Pa., and the cross-laminated paper laminate by Consolidated Water Power and Paper Co., Wisconsin Rapids, Wis.

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DISCUSSION

MR. KENNETH H. BOLLER[1] (*presented in written form*).—This paper represents the authors' pioneering efforts in establishing and maintaining a fundamental long-time creep program on plastics. Creep investigators of building materials and structural parts appreciate the problems encountered and the patience required in long-term creep testing.

We at the Forest Products Laboratory have been following Mr. Findley's work since 1944 with a great deal of interest. We have been studying the development and application of his basic equation (Eq 1) with its accompanying hyperbolic sine functions of stress to describe the constants ϵ_0 and m. We have also reviewed other types of equations and have concluded that the authors' Eq 5 also represents, to a fair degree of accuracy, the stress-time-strain relationship of tension tests of glass-reinforced plastic laminates. We find that it represents strain observations on either polyester or epoxy resin laminates that are reinforced with glass-fiber mats, straight glass fibers, woven rovings, or fabrics. Tests are currently being made in cooperation with the Navy Bureau of Ships on seven types of laminates that have had exposures up to 4 yr in conditions of 73 F and 50 per cent relative humidity atmosphere and at 73 F in water. Some tests have been made at both 0 and 45 deg to warp of a typical fabric laminate.

We find that the constants are easily equated to smooth curves that average the observed data. The values of the constants that are available on five types of laminates are presented in the accompanying Table IV for comparison with similar values in the author's Table II. As the authors have pointed out, these values may be used to compare the resistance of materials to creep. Still another use of such values is to establish curves computed from these values at various stress levels below the stress-rupture limit and for extended periods—even 10 yr—as in the authors' experiments. Such a typical family of curves is presented in accompanying Fig. 6. The strain is shown as the ordinate on a uniform scale, the time to the n^{th} power as an abscissa, and lines of constant stress are plotted to intersect the ordinate at zero time. The limit of these constant stress lines is, of course, the conventional stress-rupture curve.

MR. FRANK W. REINHART[2] (*presented in written form*).—The information presented in this paper is unique and extremely valuable. Information of this kind has been needed for a long time and the authors are to be commended for their good work and the patience that is required to follow the project for this long period.

The authors have attempted to answer or have indicated that they are aware of

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TABLE IV.—SUMMARY OF CONSTANTS DESCRIBING TENSILE STRAIN-TIME-STRESS CURVE.

$$\epsilon_t = \epsilon_0' \sinh \frac{\sigma}{\sigma_\epsilon} + m' t^n \sinh \frac{\sigma}{\sigma_m}$$

Glass-reinforced Plastic Laminates	Condition	ϵ_0', in. per in.	σ_ϵ, psi	m', in. per in.	σ_m, , psi	n
Polyester resin plus 181 fabric	Dry	0.0034	15 000	0.000445	14 000	0.090
	Wet	0.0330	80 000	0.000170	13 000	0.210
Polyester resin plus mat	Dry	0.0067	8 500	0.0011	8 500	0.190
Epoxy resin plus 181 fabric	Dry	0.0057	25 000	0.000500	50 000	0.160
	Wet	0.0250	80 000	0.000055	11 000	0.220
Polyester resin plus No. 1000 fabric	Dry	0.0015	10 000	0.000220	8 600	0.100
	Wet	0.0280	80 000	0.000108	6 500	0.190
Polyester resin plus glass woven roving	Dry	0.0180	40 000	0.00100	22 000	0.200
	Wet	0.0206	40 000	0.01460	40 000	0.230

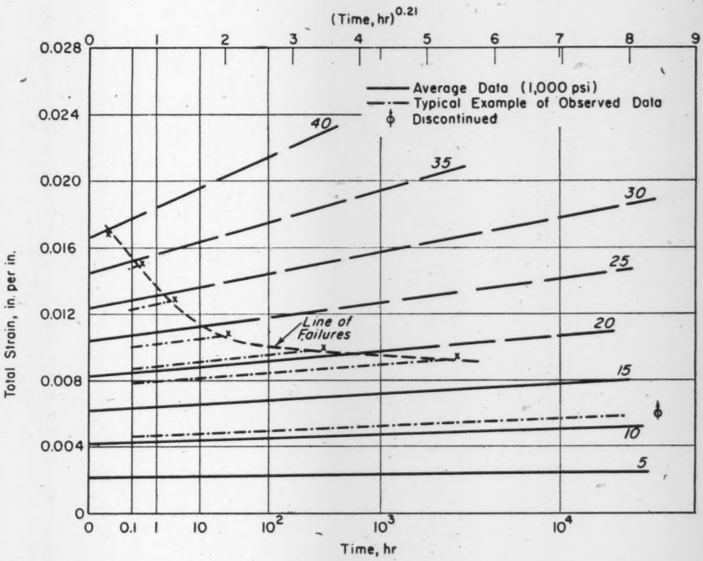

FIG. 6.—Design Curves of Average Tensile Strains of Glass-Reinforced Plastic Laminate (Polyester Resin with 181 Fabric) Tested in Water at 73 F.

many of the problems that study of their results would raise in the minds of the reviewers. One point to those interested in other types of plastics might be considered further. We wonder whether the principles developed can be used to predict, within a reasonable degree of accuracy, the creep behavior of thermoplastics. The basic differences in structure, that is, highly crosslinked resin *versus* little or no crosslinking and the presence of reinforcing materials that are fairly resistant to creep *versus* their absence, may be highly significant factors not only in the extent of creep but in the creep pattern or behavior. Some remarks by the authors on this extrapolation and on the comparison of their conclusions with those of Knowles and Dietz[3] and Schneider[4] would be helpful.

MR. E. E. THUM.[5]—The question which comes to my mind, being a metallurgist, has to do with crystalline structure of these materials. It would seem reasonable to suppose that action under creep would be closely related to the crystalline makeup—the space lattice of the atoms (or, in the case of organic compounds, the molecular arrangements).

I would hesitate very much to take data for material of one sort (plastics) and translate it to the other (metals). Can the author or anyone in the audience comment on this—recognizing of course that we know all too little about the real mechanism of creep even in metals despite the enormous amount of work which has been done.

Another comment I would like to make is that the term "high temperature" is strictly relative. What is high for one material is not high for another. You will find in the literature—and certainly, if not in the literature, then in the records of the American Steel and Wire Co.—information on a very long-time test on cold-drawn bridge wire. The chief metallurgist at the time of the Mt. Hope bridge failure in the 1920's had had in his office a 10-ft length of cold-drawn bridge wire from which was hanging a very sizable weight. He had been watching its extension (or lack of extension) for many years. I mention this to remind you of the existence of at least one bit of information about very long-time steady loading of a structural metallic material.

MR. N. BALAI.[6]—I would like to make some comment on metallic structures. We have currently three different steels in high-temperature tests: at 800 F, 900 F, and we are beginning some at 1000 F. The steels are all essentially martensitic. These steels are: (1) Crucible's Super Hytuf, (2) Universal Cyclops Thermold J tool steel, and (3) Vanadium Alloys Alloys Steel Vaseo Jet 1000. The 4000 and 5000-hr data we have currently indicate that the creep strength is alloy dependent. The Vasco Jet 1000, which is nickel-free, is substantially more creep resistant than the Universal Cyclops Thermold J which contains 1½ per cent nickel. The lean alloy steel represented by Crucible Super Hytuf is not creep resistant at all although all three steels originally were at the same hardness level of around 48 to 50 Rockwell C. The tempering temperatures of all three steels were above 1000 F in order to prevent softening of the structures during the elevated temperature tests.

MR. W. N. FINDLEY (*author*).—The remarks of the discussors are very much appreciated. It is gratifying to receive the results of additional tests reported by Mr. Boller which confirm the usefulness of the equations employed by the authors.

In answer to Mr. Reinhart's questions, the results of tension creep tests reported for the thermoplastics, polystyrene,[7] polyethylene,[8] polymonochlorotrifluoroethylene,[8] and polyvinyl chloride[8] are in good agreement with Eq 1 (and also with Eq 5). The duration of these tests was 500 to 2000 hr. Tests of thermoplastics now in progress have exceeded 10,000 hr but have not been analyzed. The tests reported indicate that the creep behavior is similar for both cross-linked and linear polymers when the total strain encountered during creep is small (less than, say, 4 per cent). For larger strains, differences in behavior may be observed. For example: in polyvinyl chloride, local flow or necking may begin; in tension, cross-linked materials fracture before large strains are encountered; and in compression[9] cross-linked materials may become much more resistant to creep as the strain approaches certain values. This is due to the effect of the primary bonds of the cross-linked structure restraining the flow.

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Regarding the comparison requested by Mr. Reinhart between the conclusions of Knowles and Dietz[3] and Schneider[4] and the conclusions of the present paper, the following may be appropriate. The Knowles and Dietz paper presents test data on methacrylate for creep, relaxation and constant strain rate tests. The duration of the creep and relaxation tests were about 0.05 hr as compared to about 90,000 hr for some of the tests reported in the present paper. The Dietz paper employs a modified Boltzmann superposition relationship for predicting creep and constant strain rate behavior from stress relaxation tests. The agreement with the data was rather satisfactory. However, Knowles and Dietz conclude in their closing statement that the "equations describe adequately only those experiments whose durations are roughly the same as that of the relaxation tests from which the values [of the constants] were obtained." The equation employed in the present paper was reasonably successful in predicting creep after 90,000 hr from data of only 2000 hr duration.

In the present paper, no comparison between creep, relaxation or constant rate testing was given. However, such comparisons employing the equation used in the present paper are given elsewhere.[10,11] The comparison of creep and relaxation is given for both a thermosetting plastic (a phenolic laminate) and a thermoplastic (poly (vinyl chloride)).[11] The agreements for these methods were also quite satisfactory as shown in the references. I do not wish to imply, however, that the Boltzmann superposition principle is unsatisfactory. As indicated previously,[12] I feel that it is the most promising principle available for predicting time dependent behavior under changing stress. It may be appropriate to recall that the method employed by Knowles and Dietz requires curve fitting to determine an expression for the relaxation curve. In the present paper the fitting was done to a creep curve.

Perhaps the main reason that the method employed by Knowles and Dietz does not predict creep accurately beyond a time equal to the duration of the relaxation tests lies in the choice of the Kohlrausch memory function. This function yields creep and relaxation functions, both of which express time in an exponential function of the form

$$(1 - e^{-a\sqrt{t}}).$$

This function has the same difficulty as that described for Eq 7 of the present paper in that the influence of time decays rapidly and the function is asymptotic to a constant (constant strain in a creep test, constant stress in a relaxation test). For the constants employed by Knowles and Dietz the stress in the relaxation test predicted by this function will be within 2 per cent of the asymptotic (constant) value in 400 sec and within 0.04 per cent within about ½ hr. Similar, but not exactly the same, conditions are predicted for creep. These predictions are not in agreement with those data on plastics from tests of longer duration which have come to my attention.

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The paper by Schneider[4] employs the same method as that by Knowles and Dietz and presents data for stress relaxation of a polyvinyl chloride for 16 hr. No data for comparison of creep and constant strain rate prediction are presented. The main conclusion drawn appears to have dangerous implications, however. It says "... by employing this equation [obtained from relaxation tests], reliable creep data can be developed more rapidly than by measuring creep under constant load over an extended period of time. In a matter of a few weeks, data can be obtained which would otherwise take several years." This conclusion is at variance with that of Knowles and Dietz. Also, whatever methods of prediction are employed, predictions are not factual data and actual test may be found to refute even the most profound theory.

Mr. Thum raised the question of the possibility of translating the data from one class of material for use in predicting the behavior of another. Such a translation is something only an engineer would attempt, of course, but an engineer must always come up with an answer using the best methods available. However, it has been found that the equation employed in the present paper is applicable to creep of some metals, at least in the so-called primary stage of decreasing creep rate. For example, creep of oxygen-free copper at room temperature[13] was found to be describable by Eq 1 as shown by the fact that a linear relationship was obtained by plotting the log of the "plastic" strain *versus* log time.

The hyperbolic sine function employed in Eq 5 was derived from the reaction rate theory and was first shown to be applicable to metals.[14] It is also identical in form to the empirical expression proposed earlier for the stress dependence of the creep rate of metals.[15]

In addition to the long-time creep test of metals mentioned by Mr. Thum, another very long-time creep test of metals was described in 1943.[16] It consisted of four tension creep specimens of a wrought nickel-chromium steel tested at 842 F for 100,000 hr. The plotted curves for these tests show that the creep rate decreased continuously for about 30,000 hr. A few years ago W. I. Mitchell and the author made an analysis of these data and found that the creep curves could be satisfactorily described by Eq 1 up to about 5000 hr when ϵ_0 was taken negligibly small. Beyond this time, the creep was greater than predicted by Eq 1. Since Robinson found from metallurgical examinations before and after the tests that banding occurred during testing, it may be that the changing structure of the material was a factor. In the analysis referred, to it was assumed that the effect of changing structure might be an exponential function of time. When this was tried it was found that the entire 100,000 hr of the creep curves could be represented reasonably well by the following equation:

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[13] E. A. Davis, "Creep and Relaxation of Oxygen-Free Copper," *Journal of Applied Mechanics*, Vol. 10, No. 2, June, 1943, p. A101.

[14] W. Kauzmann, "Flow of Solid Metals from the Standpoint of the Chemical-Rate Theory," *Transactions*, Am. Inst. Mining and Metallurgical Engrs., Inst. Metals Div., Vol. 143, p. 57 (1941).

[15] P. G. McVetty, "Creep of Metal at Elevated Temperatures—The Hyperbolic Sine Relation between Stress and Creep Rate," *Transactions*, Am. Soc. Mechanical Engrs., Vol. 65, No. 7, Oct., 1943, p. 761.

[16] E. L. Robinson, "100,000-Hour Creep Test," *Mechanical Engineering*, Vol. 65, No. 3, March, 1943, p. 166.

$$\epsilon = m'' e^{Dt} t^n,$$

where ϵ was the total strain, t the time, m'' was a function of stress, D was a constant having the value 0.000,009,94 and n was a constant independent of stress.

The data presented by Mr. Balai add another confirmation of the facts that creep resistance may be markedly affected by even slight changes in some constituents of alloys as well as small changes in environmental factors. His observations also confirm the experience of other investigators that hardness at room temperature, and for that matter other strength tests, are not necessarily indicative of creep resistance of metals.

EVALUATION OF A SINGLE-SHEAR SPECIMEN FOR SHEET MATERIAL*

By W. W. Breindel,[1] C. L. Seale,[2] and R. L. Carlson[3]

Synopsis

A single shear-type specimen which provides values of ultimate shear strength has been evaluated. As a check on the general applicability of this type of shear specimen, annealed and cold-rolled type 304 stainless steel, 2024-T3 aluminum alloy, and annealed 6Al-4V titanium alloy have been tested. The single shear-type specimen provides satisfactory results by the proper selection of the critical specimen dimensions—thickness, width, and length of shear path. Within a certain range of these variables, constant values of shear strengths can be obtained. This suggests that the value obtained is a constant for the material. Beyond the acceptable range of dimensions, buckling or tearing occurs. Proper design is achieved in specimens for which the critical loads for tearing and buckling are above that required for failure by shear.

A knowledge of the response of materials to shear stresses is of considerable interest in the design of a number of structural elements. The presence of shear stresses in shafting subjected to torsional loading presents, for example, a common design problem. No particular difficulty is, however, normally attached to this problem. The indices required for describing the response and the capacity of a given material under torsional loading can be readily obtained by the use of relatively simple test specimens and procedures. The essential features of this type of loading can be easily isolated within the specimen gage section, and the results are quite amenable to analysis and interpretation.

Another important structural element subjected to shear stresses is the shear web. Here, the shear properties of interest are those of a sheet material. The problems associated with obtaining valid experimental shear-property data for sheet materials are formidable. Ramberg and Miller (1)[4] have presented a good summary of the difficulties involved in the course of a review of methods that have been proposed. Of ten methods cited, all but two were of questionable value because the desired shear stresses could not be satisfactorily produced and isolated within a well-defined gage section.

The two methods that possessed the

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* Presented at the Sixty-first Annual Meeting of the Society, June 22–27, 1958.

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[3] Assistant Division Chief, Battelle Memorial Institute, Columbus, Ohio.

[4] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 868.

most promise involved the use of a twisted square plate (1) and an annular specimen (2). In each method, a shear-stress *versus* shear-strain curve into the plastic region can be obtained.

The above methods are of definite interest since they provide a detailed description of the response of a sheet material to increasing shear stresses. There is, however, also an interest in shear tests that provide more limited information. In these, the value or index of interest is the ultimate shear stress. In terms of design this provides, in essence, a limiting capacity or "ceiling" value.

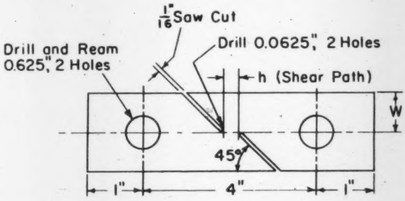

FIG. 1.—Single Shear Specimen.

By being less "demanding," this more elementary type of test can be quite simple. Ideally, it should satisfy certain requirements: it should not be based on involved assumptions or elaborate analysis, it should be possible to perform the test on standard equipment with simple fixtures.

Several shear-test methods that satisfy, at least partially, the above requirements have been proposed, and some of them have been evaluated by Fenn and Clapper (3). One of the methods evaluated makes use of the "single-shear" specimen, and it is of particular interest for sheet materials. The single-shear specimen, which is illustrated in Fig. 1,[5] was apparently first suggested by Davis *et al.* (4). Although the specimen has been used in several laboratories, there does not seem to have been any systematic evaluation of it.

The objective of the studies summarized by this paper was to provide a systematic evaluation of the single-shear specimen. The results presented consist of a general discussion of the influence of specimen dimensions on the nature of failure, and a summary of experimental data that illustrate the range of behavior possible.

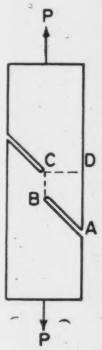

FIG. 2.—Shear Specimen.

GENERAL DISCUSSION

To provide a background against which to evaluate the data obtained in the studies summarized in this paper, a brief discussion of the nature of the loading developed in the single-shear specimen will be presented. The essential features of the single-shear specimen are represented in the drawing of Fig. 2. In Fig. 2, the dashed line between B and C represents the shear path. Ideally, failure should occur along the shear path with the remainder of the specimen remaining essentially undistorted.

To analyze the type of loading developed in the region adjacent to the shear path, it is helpful to make use of the free body outlined by the points

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[5] It is important to note that values have not been assigned to W and h. In the course of the discussion, it will be found that "proper" values of W and h vary with sheet thickness and material.

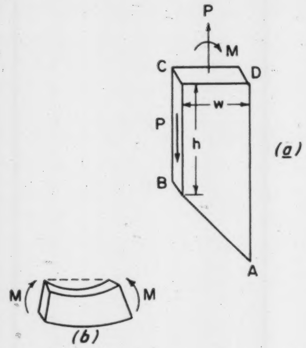

FIG. 3.—Shear Zone Free Body (*a*) and Lateral Buckling of a Beam (*b*).

TABLE I.—MECHANICAL PROPERTIES.[a]

Alloy	Material Description	Mechanical Properties				
		Direction	Elongation in 2 in., per cent	Tensile Yield Strength, psi	Ultimate Strength, psi	Elastic Modulus, psi
6Al-4V	0.095-in. sheet	Longitudinal	13.4	125 700	140 000	14.5×10^6
6Al-4V	0.095-in. sheet	Transversal	14.6	132 500	138 000	16.2
2024-T3	0.040-in. sheet	Longitudinal	18.1	54 000	72 900	10.0
304	0.050-in. sheet	Longitudinal	75	37 300	92 500	...

[a] All values as stated represent an average of 2 or 3 tests.

A, *B*, *C*, and *D*. This free body has been redrawn in Fig. 3(*a*) with the type of loading that would be developed by the external load *P*.

An inspection of Figs. 2 and 3(*a*) will indicate that two obvious modes of failure are possible. One mode of failure is in shear along the line *B-C*, as mentioned previously. It can be readily seen, however, that a second mode of failure could occur along the line *C-D*, if the dimension *W* became sufficiently small. The second failure would be a tensile-type failure that would initiate at *C* and proceed toward *D*. Initiation at *C* would be due to the tensile stress developed by the load *P* and the bending moment *M*, and the stress concentration at *C*. To avoid initiation of failure from *C* to *D*, it can be seen then, that the dimension *W* must be large enough in comparison to the dimension *h* to produce the "critical" shear stress along *C-B* before the "critical" tension is developed at *C*.

The two modes of failures noted above involve areas across *C-D* and *C-B*. Since the thickness is common for both of these areas, it would appear that critical conditions for ensuring shear failure rather than tensile failure could be developed in terms of an inequality in which it is stated that *W* must be greater than *h* by a certain amount. Experimental results to be presented in this paper will show that where these two failures are involved, this deduction is correct.

The two modes of failure discussed thus far have involved only the dimensions *W* and *h* and were independent of the thickness. A third less obvious mode of failure is due to buckling. The critical load for buckling depends not only on *h* and *W*, but also on the thickness. By referring to Fig. 3(*a*), it can be seen that if *h* and *W* are held constant but the thickness is reduced, the moment *M* can eventually be expected to become critical and to produce lateral buckling. This type of buckling (5) is more easily recognized by the similar but simpler loading illustrated in Fig. 3(*b*). It can also be illustrated by holding a piece of paper along any edge which would correspond to the side *C-B*, and applying a moment to the top edge of the paper which would correspond to the side *C-D*. The nature of the buckling obtained is then apparent.

The above discussion has indicated that failure can occur in single-shear specimens by one of three modes. The mode that will be in effect will be the one which becomes critical at the lowest load. Ideally, failure should be in shear along the plane normal to the surface of the specimen and along the axis of loading. If the tensile stresses produced in the specimen reach a critical value before the ultimate shear strength is reached, however, failure will be on a plane perpendicular to the axis of loading and across the width of the specimen. If the bending moment in the plane of the specimen becomes critical before either the critical shear or tension stress values are produced, a form of lateral buckling can be expected.

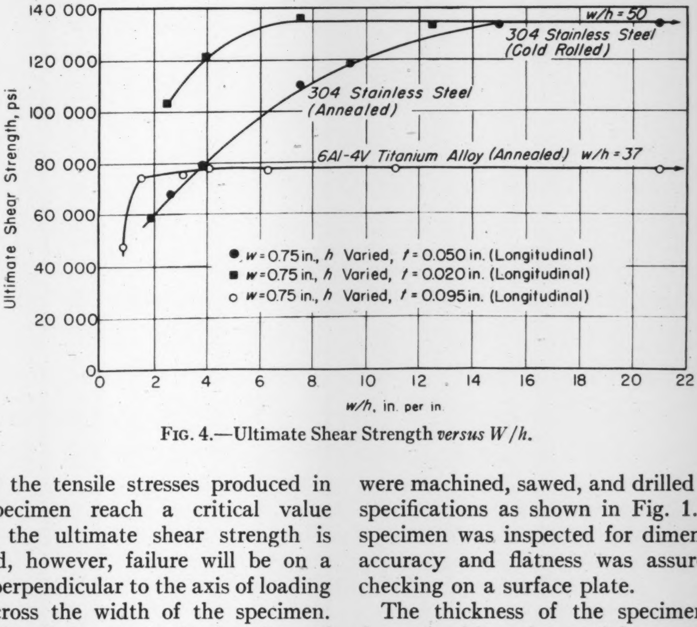

FIG. 4.—Ultimate Shear Strength *versus* *W*/*h*.

MATERIALS AND PROCEDURES

The sheet materials used in this evaluation were: titanium alloy 6Al-4V in thickness varying from 0.020 to 0.095 in.; aluminum alloy, 2024-T3, 0.020 and 0.040 in. thick; stainless steel type 304, 0.020 and 0.050 in. thick. The stainless steel was evaluated in the annealed and cold-rolled conditions and the titanium alloy was tested only in the annealed state. All specimens of a given alloy were obtained from the same sheet. The mechanical properties are given in Table I.

The specimen blanks were cut from a flat sheet with the desired orientation with the rolling direction. The blanks were machined, sawed, and drilled to the specifications as shown in Fig. 1. Each specimen was inspected for dimensional accuracy and flatness was assured by checking on a surface plate.

The thickness of the specimen was determined by using a minimum of three micrometer readings within the region of shear and the average recorded. The length of the shear path was measured on both faces by means of an optical comparator.

Tests were performed on a universal hydraulic testing machine. The loading shackles were of the self-aligning type and consisted of hardened and ground steel plates in double shear. The head travel was kept constant at 0.03 in. per min. All tests discussed in this paper were performed at room temperature.

In the discussion to follow, the ultimate shear strength is defined as the maximum load divided by the shear area (length of shear path, h, times the sheet thickness, t). In all tests, it was observed that the fracture load was equal to or less than the ultimate load.

DISCUSSION OF RESULTS

Three modes of failure were described earlier in this paper. In the discussion to follow, test data will be used to demonstrate the manner in which transitions from shear to tearing failures and shear to buckling failures occur.

The results on the annealed 6Al-4V titanium alloy as shown in Fig. 4 provide an example of a transition from shear to tearing failure. In these tests t and W were held constant and h was varied. For W/h values greater than about 2.5, the specimens failed by shearing, and the value of the ultimate shear strength was constant. For W/h values less than 2.5, however, the specimens failed by tearing. The result of a transition from satisfactory shear failure to unsatisfactory failure by tearing is clearly illustrated by this plot. The "critical" load for tearing drops below the "critical" load for shear at about $W/h = 2.5$.

The third possible mode of failure is by buckling. As sheet thickness is decreased, it should be possible to observe a transition from failure by shearing to failure by buckling. Specimens were, therefore, prepared from the same sheet of 6Al-4V to illustrate the third mode of failure-buckling. In this series of tests the W and h values were held constant and t was varied. Figure 5 illustrates the results of this experiment. The fixed value of $W/h = 3.3$ was found to be satisfactory only for thicknesses greater than about 0.025 in.[6] For thinner specimens, the more complicated failure by buckling was observed.

To illustrate also the change in the resistance to buckling of variations in the value of W/h, the dashed curves of Fig. 5 have been included. By increasing the W/h value, the resistance to buckling would be increased. Satisfactory shear tests could, therefore, be conducted on sheet thinner than 0.025 in. If, however, the W/h value were decreased, the resistance to buckling would decrease and satisfactory shear tests could be conducted only for thicknesses greater than 0.025 in.

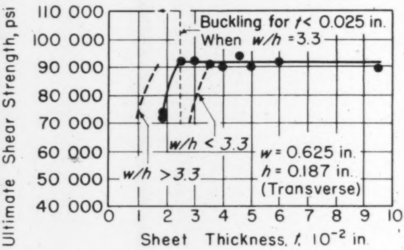

FIG. 5.—Ultimate Shear Strength *versus* Sheet Thickness for Annealed 6Al-4V Titanium Alloy.

In considering failures by buckling, it is important to emphasize that this mode of failure may be confused with failure by tearing. The two modes are distinct, however. In tearing, the sheet remains essentially in its original plane. During a buckling failure, the corner at point A of Fig. 2 tends to come out of the original plane of the sheet. If the shear specimen is strained after the buckling load is reached, a tearing action can initiate at point *B* or *C*. Tearing in this instance, however, occurs after buckling.

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[6] It should be noted that the ultimate strength indicated in Fig. 5 is about 92,000 psi, whereas a value of about 78,000 psi was indicated in Fig. 4. These specimens were, respectively, for the transverse and longitudinal directions. A directionality of properties is also indicated for this material in Table I.

The three modes of failure have also been observed in tests on single-shear specimens of the aluminum alloy 2024-T3. The results of Fig. 6 indicate that a transition from shearing to tearing occurs at W/h less than about 2.5 for the 0.040-in. sheet. For 0.020-in. sheet, a transition from shearing to buckling occurs at about $W/h = 4$. This again illustrates that the operative mode of failure is determined by the dimensions t, h, and W. It is important also to note again that the ultimate shear strength is essentially constant for satisfactory shear failures.

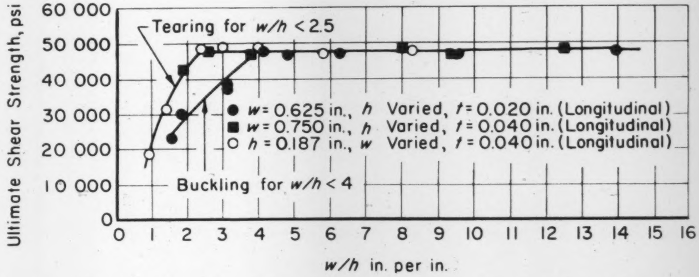

Fig. 6.—Ultimate Shear Strength *versus* W/h for 2024–T3 Aluminum Alloy Sheet.

When plastic behavior is involved in a buckling problem, the work-hardening characteristics of the specimen material are a factor in determining critical conditions. To illustrate this effect, results for annealed and cold-rolled (60 per cent reduction) 304 stainless steel are shown in Fig. 4. As can be seen, a leveling off of the curve appears at about $W/h = 7$ for the cold-rolled stainless steel. For values less than about $W/h = 7$, failure occurred by buckling. For the annealed stainless steel, a value of about $W/h = 17$ was achieved before a tendency toward a constant value was realized. For W/h values less than about 17, buckling failures were observed. The increase in the resistance to buckling is even more impressive when it is noted that it was achieved with a marked decrease in thickness. This, in the absence of cold working, would have a tendency to reduce the resistance to buckling (see, for example, the curves of Fig. 5). The results, therefore, suggest that for a type 304 stainless steel specimen of hardness equivalent to the cold-rolled specimens, but of a thickness of, say, 0.040 to 0.090 in., it would be expected that constant values of ultimate shear strength would be obtained for values of W/h less than 7.

In conclusion, it must be emphasized that the specimen of Fig. 1 with fixed values of W and h cannot be regarded as a standard specimen. It is apparent from the results that values of W and h that will result in satisfactory results depend on the sheet thickness and the plastic properties of the material.

Summary

The studies summarized by this paper indicate that the single-shear specimen evaluated can, if properly designed, provide ultimate shear-strength values for sheet material. Proper design can be achieved by the use of specimen dimensions which raise the critical loads for tearing and buckling above that required for failure by shear.

Data on several materials illustrates the manner in which transitions from satisfactory shear failures to tearing and buckling failures occur with dimensional changes. Tearing failures can be avoided by the use of W/h values greater than about 2.5. Buckling failures are more complex and depend on the thickness, t, and the work-hardening characteristics, as well as W and h. The occurrence of buckling failures in a given single-shear specimen can be eliminated by a sufficient increase in either the W/h value or the sheet thickness.

Acknowledgment:

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A HIGH DAMPING MAGNESIUM ALLOY FOR MISSILE APPLICATIONS*

By G. F. Weissmann[1] and W. Babington[1]

Synopsis

It is of greatest importance for the proper functioning of the components of guided missiles that resonance vibrations occurring in the structural members of the missile be kept at a permissible level. The use of materials of high damping capacity may contribute considerably toward the reduction of resonance amplifications in the missile structure and its components.

Damping studies were conducted on several magnesium alloys, employing flat cantilever specimens stressed in bending. The specific damping capacity of the alloys was established as a function of the strain, considering the strain distribution of the test specimen. The resonance amplification factor of the cantilever specimen was determined analytically and then compared with the results of vibration tests. One magnesium alloy containing nominally 0.6 per cent zirconium showed a very high damping capacity, combined with adequate mechanical properties. The effect of varying zirconium contents, of heat treatments, and machining were investigated. The damping of the alloys in both sand-cast and die-cast conditions was compared.

The housing, instrument plate and gyro mounting brackets for the main guidance section of a guided missile have been satisfactorily sand cast, using this high damping alloy. It is further proposed to die cast the instrument plate of the same alloy.

Resonance amplitudes and accelerations are of considerable importance for the proper functioning of electronic components of guided missiles. These accelerations can be substantially reduced if the excited forces are kept low. This can frequently be attained by selecting structural materials with sufficiently high internal damping, that is, materials which will absorb a high percentage of the excited vibrational energy.

Early in the redesign of the guidance unit of a missile, the need for such high damping structural materials was recognized, in order to afford the maximum protection from damaging vibration for the electronic components of this unit. In the interest of weight reduction, the use of magnesium alloys for structural parts was indicated. Conventional magnesium materials such as the AZ81A alloy were originally proposed for the main housing of the guidance unit. Vibration tests have shown that excessive resonance amplification may occur when employing such alloys. This led to an

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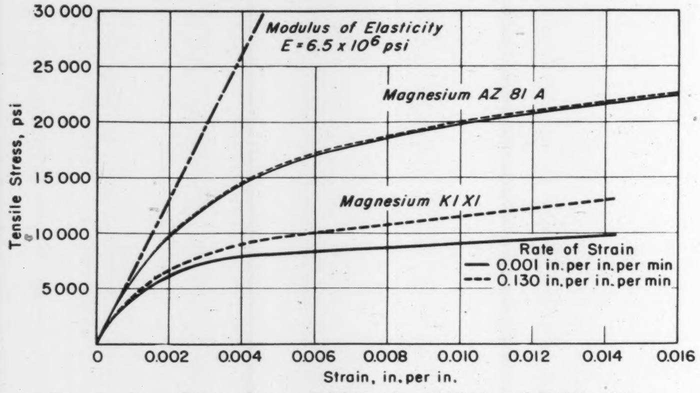

FIG. 1.—Stress-Strain Curve of Magnesium AZ81A and K1X1 Alloys.

TABLE I.—TENSILE PROPERTIES OF SOME MAGNESIUM ALLOYS.[a]

Material Designations	Yield Strength,[b] psi, 0.2 per cent Offset	Tensile Strength, psi	Elongation in 2 in., per cent
SAND CAST—AS CAST			
Commercial magnesium	4 100	14 500	6.8
Magnesium-zirconium (0.4 per cent zirconium)	5 800	22 400	21.2
Magnesium-zirconium K1X1 (0.6 per cent zirconium)	7 900	24 000	21.9
Magnesium-zirconium (1.0 per cent zirconium)	7 600	22 500	16.4
Magnesium-silicon S1X1 (0.75 per cent silicon)	8 600	13 300	1.0
Magnesium-aluminum-zinc,[c] AZ81A	14 600	24 800	2.5
DIE CAST—AS CAST			
Commercial magnesium	7 100	20 400	9.4
Magnesium-zirconium K1X1	10 700	22 800	8.7
Magnesium-aluminum-zinc[c] AZ91B	21 100	26 700	1.5

[a] All values represent average of at least 4 tests.

[b] A modulus line of 6.5×10^6 psi was constructed tangent to the stress-strain curve and 0.2 per cent offset yield strengths were determined from this line and reported.

[c] Commercial alloys.

investigation of available materials possessing superior damping properties.

HISTORICAL BACKGROUND

The unusually high damping capacity of certain magnesium alloys has been recognized for some time (**1**,**2**).[2] However, the practical significance and possible application of these data was not appreciated; consequently development of high-damping magnesium alloys has been somewhat neglected. The extremely rapid growth of the missile industry and the search for better materials to fulfill specific applications has focussed strong attention on the use of structural materials possessing adequate strength and light weight combined with high damping capacity. Early work at the Dow Chemical Co. had shown that the damping capacity of certain magnesium alloys was considerably better than others and more specifically that an alloy of magnesium plus 0.75 per cent silicon possessed a damping capacity far superior to any other then known structural light alloy. Later work proved the validity of these earlier results. Because of inherent difficulties in casting and fabricating the magnesium-silicon alloy, a binary alloy of magnesium plus 0.6 per cent zirconium was developed. This new alloy, designed as K1X1, possesses an even better damping capacity than the magnesium-silicon alloy. The finer grain size of K1X1 alloy results in higher strength and ductility and considerably reduces problems related to casting and fabrication. The fact that the alloy has a relatively narrow freezing range may lead to some problems concerning castability and shrinkage. It is expected, therefore, that certain shapes will be somewhat more difficult to cast in K1X1 alloy than in the commercial magnesium-aluminum-zinc alloys. Satisfactory castings by both sand-and die-casting practices have been produced.

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[2] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 885.

TABLE II.—MATERIALS TESTED AND TEST CONDITIONS.

Designation	Material	Surface	Heat Treatment, deg Fahr	Finish
A......	Mg—commercial, sand cast	As cast[a]	...	...
B......	Mg—commercial, sand cast	Machined	...	...
C......	Mg—commercial, sand cast	Machined	16 hr at 600	...
D.....	Mg + 0.2 % Zr, sand cast	As cast	...	...
E......	Mg + 0.4 % Zr, sand cast	As cast	...	...
F......	Mg + 0.6 % Zr, sand cast	As cast	...	...
G.....	Mg + 1.0 % Zr, sand cast	As cast	...	...
1.....	Mg + 0.6 % Zr, sand cast	Machined	...	...
2.....	Mg + 0.6 % Zr, sand cast	As cast	16 hr at 200	...
3.....	Mg + 0.6 % Zr, sand cast	As cast	16 hr at 400	...
4.....	Mg + 0.6 % Zr, sand cast	As cast	16 hr at 600	...
5.....	Mg + 0.6 % Zr, sand cast	As cast	15 min at 200	...
6.....	Mg + 0.6 % Zr, sand cast	Machined	15 min at 200	...
7.....	Mg + 0.6 % Zr, sand cast	As cast	15 min at 200	Modified Dow 17
8.....	Mg + 0.6 % Zr, sand cast	Machined	15 min at 200	Modified Dow 17
9.....	Mg + 0.6 % Zr, sand cast	As cast	15 min at 200	Electroless nickel
10.....	Mg + 0.6 % Zr, sand cast	Machined	15 min at 200	Electroless nickel
11.....	Mg + 0.6 % Zr, sand cast	Cut from housing wall	...	...
12.....	Mg + 0.6 % Zr, sand cast	As cast	3 months, aging	...
13.....	Mg + 0.6 % Zr, die cast	As cast	...	...
a......	Mg-AZ91B, die cast	As cast	...	...
b......	Mg-AZ81A, sand cast	As cast	...	...
c......	Mg + 0.75 % Si, sand cast	As cast	...	...
d......	Mg + 0.75 % Si, sand cast	Machined	...	...
e......	Mg + 0.75 % Si, die cast	As cast	...	...

[a] All samples in the "as-cast" condition have been sandblasted and pickled.

Mechanical Properties

The mechanical properties of the high damping magnesium alloy K1X1 have been compared with those of a conventional magnesium alloy AZ81A. Figure 1 shows the stress strain curves for these alloys. As shown in this figure, the rate of strain has no effect on the tension test results of the AZ81A alloy. However, the K1X1 alloy shows higher values for both yield strength and tensile strength, at higher rates of strain. This phenomenon is an indication of a high inherent damping capability of the material. The proportional limit cannot be established for the K1X1 alloy due to the continuous curvature of the stress-strain curve. The 0.2 per cent offset yield strength has been established by measuring the deviation of the actual stress-strain curve from the artificial stress-strain relation based on a constant modulus of elasticity of $E = 6.5 \times 10^6$ psi. The mechanical properties of magnesium-zirconium alloys in the sand- and die-cast conditions, as well as the mechanical properties of the AZ81A sand cast alloy and the AZ91B die cast alloy are shown in Table I. The yield strength of the die cast K1X1 alloy is considerably higher than that of the sand cast alloy.

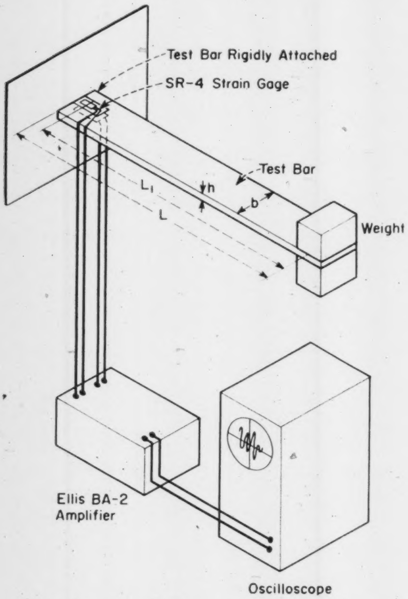


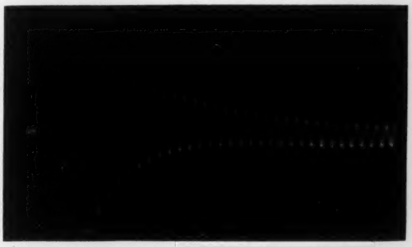
FIG. 2.—Block Diagram of Apparatus.

DAMPING PROPERTIES

The literature on damping is notable for lack of agreement between different investigators. Comparison of results is made more difficult because of the many terms that have been used to express material damping: modulus of decay (relaxation time); logarithmic decrement; half-band width; energy absorption per cycle (damping energy); energy absorption per cycle per unit volume (specific damping energy); relative energy absorption per cycle (damping capacity); relative energy absorption per cycle per unit volume (specific damping capacity); viscosity; complex modulus of elasticity; and wave propagation constants. The difficulty of evaluating damping tests by different investigators is increased, furthermore, by the presence of different nonuniform strain distributions of the test specimens. Frequently, the shape of the test specimen and the stress level at which the damping tests have been conducted is not available for evaluation.

In general, the internal damping of a complicated structural part may be determined in two ways: (*a*) The particular structural part may be used as a specimen and tested under conditions analogous to the operating conditions. This method is costly and often even impossible to carry out; (*b*) The damping may be measured in a conventional manner using a simple shaped specimen. The results of this test are then used to predict the damping of the complicated member. The latter procedure undoubtedly is superior to the first provided the data of the conventional test can be interpreted in a simple manner. Several methods for evaluation of the damping tests have been presented. E. R. Podneiks and B. J. Lazan (**3**) show a comprehensive method for determining the specific damping energy, considering the stress distribution. The specific damping energy is expressed in their work as a function of the stress. G. Denkhaus (**4**) determines the specific damping capacity from vibration tests conducted in bending, using the strain as a parameter. The latter procedure has been followed in this work.

Tests have been conducted to compare the damping capability of the high damping magnesium alloy K1X1 with a conventional magnesium alloy such as the AZ81A. The effect of zirconium content, heat treatment, machining, and finish has been investigated.



(*a*) Magnesium K1X1 alloy, sand cast; time: 1 unit = 0.1 sec; strain: 1 unit = 50×10^{-6}.

(*b*) Magnesium AZ81A alloy, sand cast; time: 1 unit = 0.1 sec; strain: 1 unit = 50×10^{-6}.

FIG. 3.—Decay of Free Vibration of Cantilever Beam for Magnesium K1X1 and Magnesium AZ81A Alloy.

The damping properties of many of the test specimens are shown in terms of the logarithmic decrement of a cantilever beam with a weight attached to its free end, which gives a good comparison between the damping of the different alloys and the effect of different conditions. For the more promising alloys the specific damping capacity has been determined experimentally. The resonance amplification factor and the maximum fiber strains of the same cantilever beam specimens at resonance have been calculated. The results have been compared with actual vibration measurements on these cantilever beams. Finally, measurements have been made on the cast housing for the main guidance section of the missile. The relative improvement obtained by using a high damping alloy compared to a conventional magnesium alloy has been calculated and compared with results obtained on the vibration table.

MATERIALS TESTED

All of the magnesium alloy specimens were furnished by the Dow Chemical Co. The specimens were supplied in the form of 9 in. long bars with cross-sectional dimensions of $1\frac{1}{2}$ by $\frac{1}{4}$ in. Table II shows the tested magnesium alloys, their surface conditions, heat treatments, and finishes.

TEST METHOD

The test method used for determination of the damping capabilities of the various materials investigated is shown in Fig. 2. One end of the specimen was clamped rigidly in a heavy Baldwin testing machine. An arbitrarily selected weight of 340 g was fastened to the other end of the specimen. SR-4 strain gages were attached as closely as possible to the fixed end of the specimen. The strain gages were mounted on opposite sides of the beam. Both were used as active gages in a Wheatstone bridge circuit, and hence the output potential was twice as great as when but one active gage was employed. The strain gages were connected to an Ellis BA-2 amplifier which in turn was connected to an oscilloscope. After the specimen had been set in motion by tapping the weight with a light hammer, the wave trace was observed on the oscilloscope. Pictures of the wave trace were taken by means of an oscilloscope camera using polaroid film. Figure 3 shows the wave traces of the magnesium alloy containing 0.6 per cent zirconium (No. F of Table II) and the magnesium alloy AZ81A (No. b of Table II). The oscilloscope had been previously calibrated in terms of strain. The abscissa represents time, the ordinate fiber strain values. Similar photographs have been taken of all the different magnesium alloys listed in Table II. Different amplifications have been used for obtaining wave traces at different strain levels.

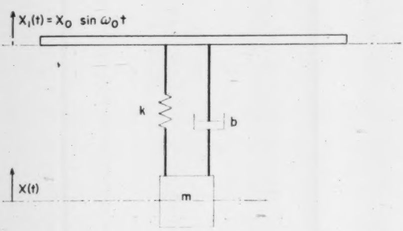


FIG. 4.—Linear One-Degree of Freedom Oscillator.

TEST RESULTS

The test apparatus is a linear one-degree of freedom oscillator which can be symbolized by a mass-spring-dashpot system as shown in Fig. 4. The equation of motion for this system is

$$m\ddot{x} + b(\dot{x} - \dot{x}_1) + k(x - x_1) = 0 \quad (1)$$

where:

m = mass of system, lb-in.$^{-1}$ sec^2,
k = spring constant, lb-in.$^{-1}$,
b = damping coefficient, lb-in.$^{-1}$ sec,
x = displacement of mass m, in.,
$x_1 = x_0 \sin \omega_0 t$ = displacement of base, in.,
$x(t)$ = amplitude of mass, m, in.,
$x_1(t)$ = forced amplitude of base system, in.
x_0 = amplitude of base, in., and
ω_0 = circular frequency, cps.

Two solutions of this equation are of particular interest:

(*a*) the case where $x_0 = 0$ (corresponding to free oscillation), and
(*b*) the case where x_0 = constant (corresponding to forced vibrations).

The logarithmic decrement and the damping capacity of the test specimens are derived from case (*a*) and are obtained by direct measurements of the amplitudes or values proportional to the amplitudes such as stress or strain. They are used as a measurement of the damping capability of the tested specimen. The logarithmic decrement has been calculated by means of:

$$\bar{\delta} = \frac{1}{n} \log \frac{\epsilon_m}{\epsilon_{m+n}} \quad (2)$$

where:

$\bar{\delta}$ = logarithmic decrement of test specimen,
ϵ_m = m^{th} measured strain amplitude,
ϵ_{m+n} = $(m + n)^{\text{th}}$ measured strain amplitude, and
n = number of cycles between measured amplitudes.

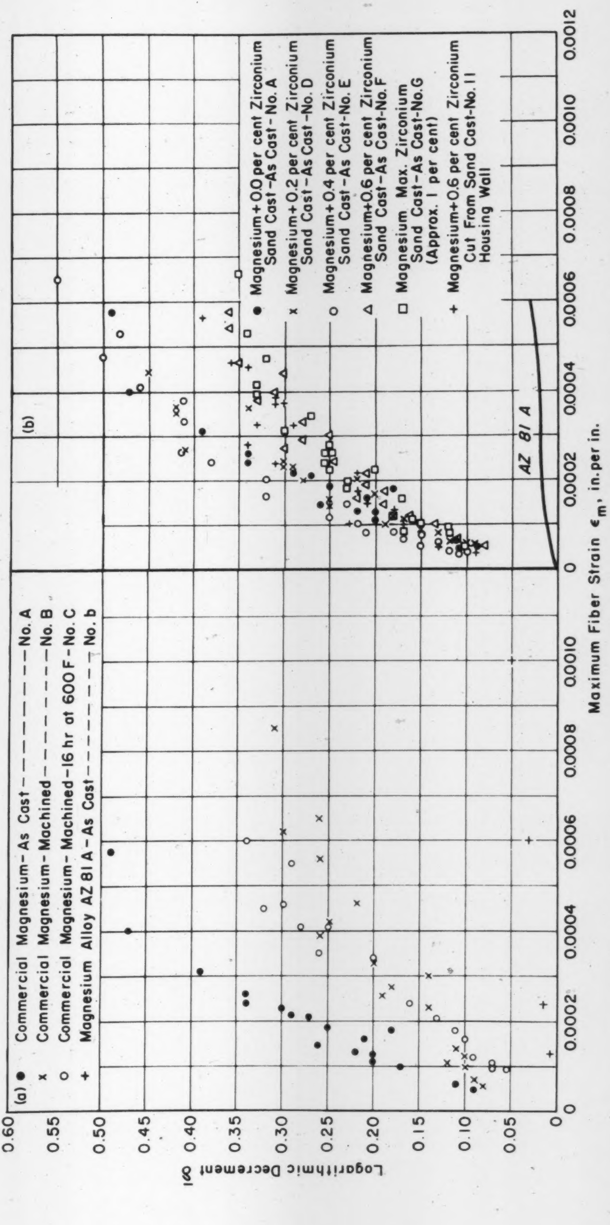
The damping capacity of the vibrating system is defined as the ratio of the absorbed energy per cycle to the maximum elastic energy and can be determined by strain measurements from:

$$\bar{\Psi} = 1 - \left(\frac{\epsilon_{m+1}}{\epsilon_m}\right)^2 \quad (3)$$

where $\bar{\Psi}$ = damping capacity of test specimen.

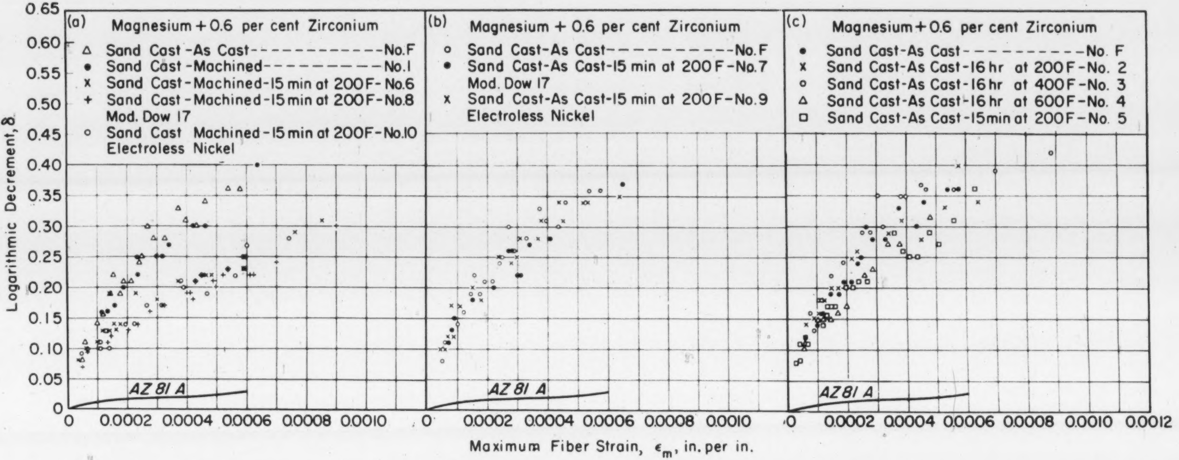
The relationship between the damping capacity and the logarithmic decrement follows from Eqs 1 and 2

$$\bar{\Psi} = 1 - e^{-2\bar{\delta}} \quad (4)$$



(a) Commercial magnesium specimens. (b) Magnesium specimens—effect of zirconium content.

FIG. 5.—Logarithmic Decrement.



(*a*) Machined specimens.

(*b*) Effect of finish, as cast specimens.

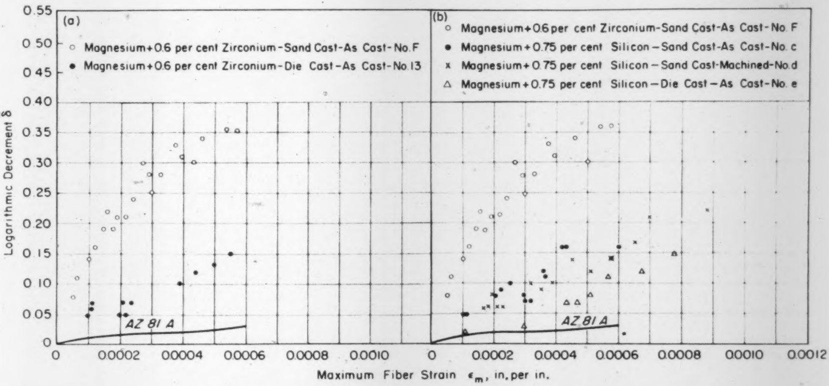
(*c*) Effect of heat treatment, as cast specimens.

FIG. 6.—Effect of Heat Treatment and Finish on the Logarithmic Decrement of K1X1 Specimens.

For small values of the logarithmic decrement this equation simplifies to:

$$\bar{\Psi} = 2\bar{\delta} \quad \text{if} \quad \bar{\delta} \ll 1 \ldots\ldots (4a)$$

For specimens with low damping capabilities the difference in the strain amplitude over 5 cycles ($n = 5$) has been used to determine the logarithmic decrement $\bar{\delta}$. For specimens with high damping, however, the difference between two successive amplitudes ($n = 1$) has been used for the calculation.



(*a*) Sand-cast and die-cast K1X1 specimens. (*b*) S1X1 and K1X1 specimens.

FIG. 7.—Comparison Between the Logarithmic Decrement.

The logarithmic decrements of all the samples listed in Table II have been calculated and are shown in Figs. 5 to 7. They have been plotted as functions of the maximum fiber strain. The maximum fiber strain has been obtained from the measured strain values by means of the following relationship:

$$\epsilon_{\max} = \frac{L}{L_1}\,\epsilon_{\text{measured}}$$

L and L_1 are shown in Fig. 2.

Figure 5(*a*) shows the logarithmic decrement of the commercial magnesium samples, in the as-cast, machined, and machined and heat treated conditions. These data show that machining reduces the damping capability of commercial magnesium.

Figure 5(*b*) shows the effect of the zirconium content on the logarithmic decrement of the test specimens. An increased zirconium content slightly reduces the damping properties; however, it improves the mechanical properties of the alloy (Table I). Approximately 0.6 per cent zirconium content appears to provide the optimum combination of damping and mechanical properties.

Figure 6(*a*) shows the effect of machining and subsequent heat treatment of the K1X1 test specimens. Machining alone does not reduce the damping capability of the material. However, an additional heat treatment reduces the logarithmic decrement. It will also be noted from these data that neither the Modified Dow 17 nor the Electroless Nickel finishes on machined specimens have any additional effect on the logarithmic decrement of the test specimens. Figure 6(*b*) shows the effect of the finish on the damping capability of the unmachined

K1X1 specimens. Again, no significant change could be observed.

Figure 6(*c*) shows the effect of heat treatment on the as-cast K1X1 specimens. No significant reduction of the damping has been observed as a result of these heat treatments.

Figure 7(*a*) compares the logarithmic decrements of the K1X1 alloy in the sand-cast and die-cast conditions. The die-cast specimens showed a considerably lower logarithmic decrement than sand-cast specimens.

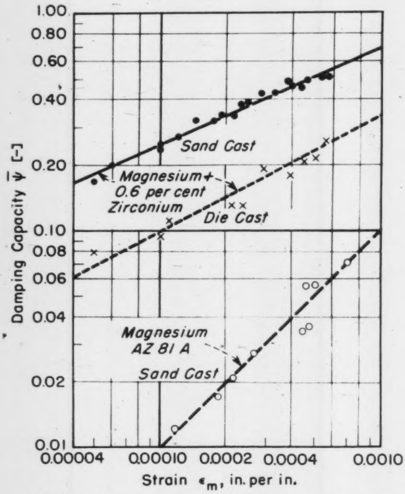


FIG. 8.—Damping Capacity of Cantilever Beam.

Figure 7(*b*) compares the logarithmic decrement of a K1X1 sample with a specimen containing magnesium–0.75 per cent silicon. It will be noted that the K1X1 alloy showed considerably higher damping than the magnesium-silicon alloy.

K1X1 specimens have been tested again after 3 months aging and no difference in damping has been observed. The damping capacity of the magnesium alloy (K1X1) containing 0.6 per cent zirconium in the sand-cast (No. *F* of Table I) and die-cast (No. 13 of Table I) conditions, as well as for the conventional magnesium alloy AZ81A has been calculated by means of Eqs 3 or 4, for the K1X1 and by means of Eq 4*a* for the AZ81A.

The values of the damping capacity $\bar{\Psi}$ have been plotted on a log-log scale as a function of the maximum fiber strain ϵ_m and are shown in Fig. 8. It appears that the relationship betewen damping capacity and maximum fiber strain can be represented by a straight line on the log-log scale which means also that the damping capacity $\bar{\Psi}$ of the specimens can be expressed as the following function of the fiber strain

$$\bar{\Psi} = \bar{\alpha}\epsilon_m\bar{\beta} \quad \text{(5)}$$

$\bar{\alpha}$ and $\bar{\beta}$ are constants depending on the material and the shape and size of the test specimens. $\bar{\alpha}$ and $\bar{\beta}$ have been calculated using the method of least squares and are shown in Table III.

TABLE III.—DAMPING CAPACITY $\bar{\Psi}^a$ AND SPECIFIC DAMPING CAPACITY Ψ^b AS A FUNCTION OF THE MAXIMUM STRAIN ϵ_m OF TEST SAMPLE.

Numbers of Table II	Material	$\bar{\alpha}^c$	$\beta = \bar{\beta}^c$	α^c
F.....	Mg + 0.6 % Zr, sand cast, as cast	14.53	0.44	19.1
13....	Mg + 0.6 % Zr, die cast, as cast	13.07	0.53	18.1
b.....	Mg-AZ81A, sand cast, as cast	100.5	1.01	179.0

[a] $\bar{\Psi} = \bar{\alpha}\epsilon^{\beta}$.

[b] $\Psi = \alpha\epsilon^{\beta}$.

[c] $\bar{\alpha}$ and $\bar{\beta}$ are constants depending on the material and the shape and size of the test specimen, α and β are constants depending only on the material.

RELATIONSHIP BETWEEN DAMPING, STRAIN MAGNITUDE AND STRAIN DISTRIBUTION

It is desirable to determine the mathematical relationship between damping and strain magnitude, strain distribution, previous strain history, and temperature. The previous strain history affects various materials differently. It is therefore impossible to generalize strain history effects at this time and to express such effects mathematically. It is also impossible to generalize temperature effects at this time. However, there are sufficient data (**4,6,7**) to indicate that for many materials the effect of the strain magnitude (at prescribed constant temperature, strain history and, uniform strain distribution) may be expressed by the equation

$$\psi = \alpha \epsilon^{\beta} \quad \text{.............} (6)$$

where:

ψ = specific damping capacity,

ϵ = strain, and

α, β = material constants.

The specific damping capacity ψ has been defined as the ratio of the specific damping energy ΔU to the specific elastic energy U

$$\psi = \frac{\Delta U}{U} \quad \text{.............} (7)$$

The specific damping capacity is the damping capacity of a unit volume subjected to a uniform strain distribution.

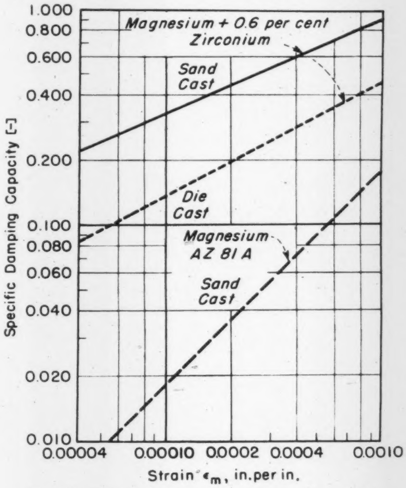


FIG. 9.—Specific Damping Capacity of Magnesium Alloys.

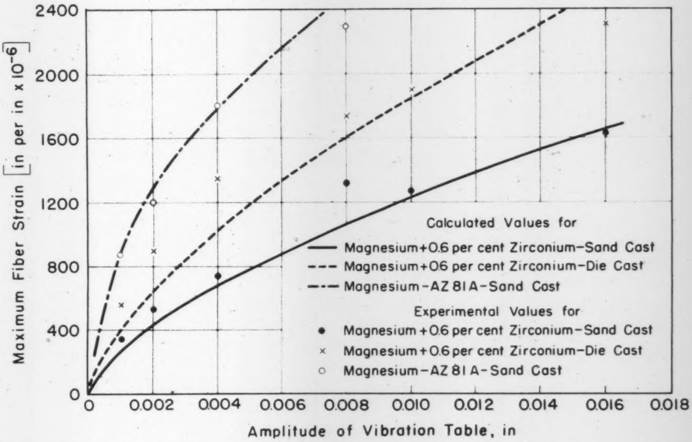
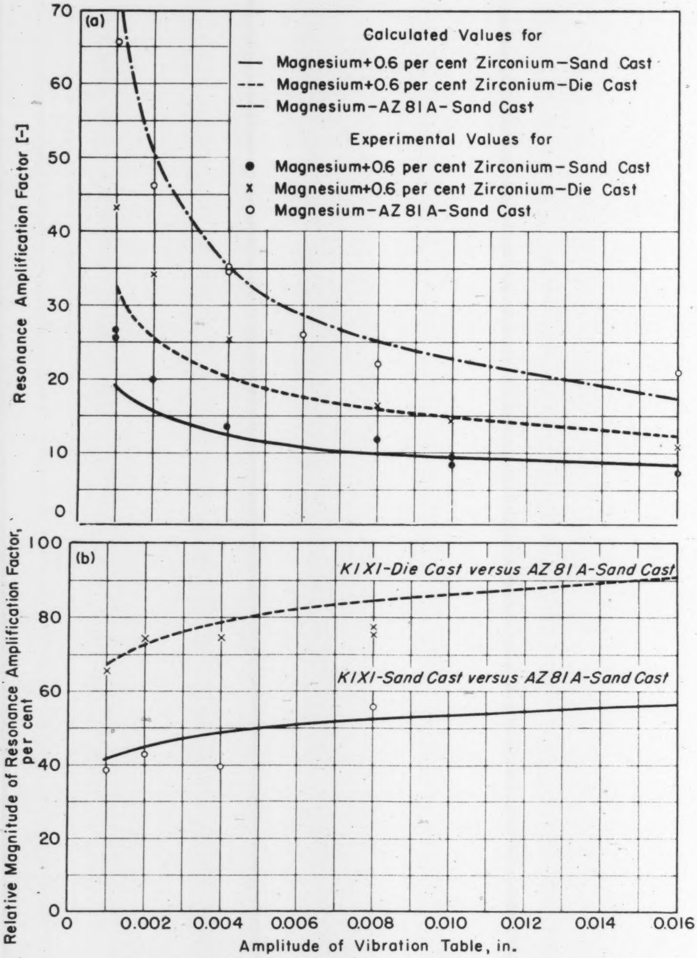


FIG. 10.—Calculated and Measured Maximum Fiber Strains of Cantilever Specimens at Resonance.



(*a*) Calculated and measured. (*b*) Relative magnitude.

FIG. 11.—Resonance Amplification Factors of Cantilever Specimens.

In most engineering structures and also in many specimens used for damping measurements, the strain distribution is not uniform. The relationship between the specific damping capacity ψ of the material and the damping capacity $\bar{\psi}$ of a structure or specimen subjected to bending can be derived mathematically. The mathematical relationship between the specific damping capacity ψ and the damping capacity $\bar{\psi}$ of a vibrating cantilever beam has been derived in the appendix. The results show that:

$$\bar{\psi} = \psi \left(\frac{3}{3 + \beta)}\right)^2$$

$$= \bar{\alpha}\epsilon^{\bar{\beta}} = \alpha \left(\frac{3}{3 + \beta}\right)^2 \epsilon^{\beta} \quad \text{..(8)}$$

from which follows:

$$\bar{\alpha} = \alpha \left(\frac{3}{3 + \beta}\right)^2 \quad \text{.........(9)}$$

$$\beta = \bar{\beta}$$

By means of Eqs 8 and 9, it is now possible to determine the damping capacity of a vibrating cantilever beam from a known specific damping capacity or to calculate the specific damping capacity from data obtained by vibration tests of a cantilever beam. The specific damping capacities obtained for the K1X1 alloy in the sand- and die-cast conditions as well as for the AZ81A sand-cast alloy are shown in Fig. 9. Table III shows the material constants α and β necessary to calculate the specific damping capacity as a function of the strain amplitude.

THE RESONANCE AMPLIFICATION FACTOR

From the known specific damping capacity $\psi = \alpha\epsilon^{\alpha}$ it is now possible to calculate the maximum strain in forced vibration at resonance and the resonance amplification factor of the cantilever beam. Based on the equation of motion for forced vibration (Eq 1, case (*b*)), it can be shown that:

$$A_r = \frac{A_m}{x_0} = \frac{2\pi}{\bar{\psi}} \quad \text{.........(10)}$$

where:

A_r = resonance amplification factor,
A_m = amplitude of mass of vibrating system,
x_0 = amplitude of base of vibrating system, and
$\bar{\psi}$ = damping capacity of vibrating system.

The maximum fiber strain and the resonance amplification factor of a vibrating cantilever beam have been derived mathematically as a function of the amplitude of the base of the vibrating system. The derivation is shown in the appendix.

The computed strain values are shown in Fig. 10. They are plotted as functions of the amplitudes of the base. Experimental results obtained by mounting the same test specimens as were used in the free vibration tests on a vibration table are also shown in Fig. 10.

Figure 11(*a*) shows both the calculated resonance amplification factors and the experimental test results. The agreement of the calculated and experimentally determined values, in both cases, is considered very good. The greatest differences between calculated and measured values occur at the lower strain levels. Free vibration tests conducted with the same specimens as were used in the forced vibration tests show some decrease of the specific damping capacity of the material in comparison to the values obtained originally. This indicates that previously applied large strains cause a reduction of the damping capabilities of the material.

Following a similar procedure, it is possible to calculate the damping capacity, maximum fiber strain at resonance, and resonance amplification factor of a structure subjected to a different moment distribution from the specific damping capacity of a material. It is also possible to determine the specific damping capacity from the experimentally determined values of the damping capacity, maximum fiber strain at resonance or the resonance amplification factor of a structure subjected to a different moment distribution.

COMPARISON OF RESONANCE AMPLIFICATION FACTORS

In many engineering applications it is sufficient to know the relative magnitude of the resonance amplification factor obtained by changing a structure from a conventional alloy to a high damping alloy. The ratio is:

$$R = \frac{A_{r1}}{A_{r2}} 100 \text{ [per cent]} \ldots\ldots (11)$$

where:

A_{r1} = resonance amplification factor for high damping material, and

A_{r2} = resonance amplification factor for conventional material.

If the resonance amplification factor A_{r2} of a structure made of a conventional alloy has been determined by actual measurement, it is possible to predict the magnitude of the resonance amplification factor of the same structure made of a high-damping alloy.

The relative magnitude of the resonance amplification factor of the K1X1 alloy cantilever beam in the sand- and the die-cast conditions, to the cantilever beam tests made on the conventional AZ81A alloy has been computed by means of Eq 11 and is shown in Fig. 11(*b*), together with the experimentally determined values. The agreement between these two sets of values is considered excellent.

HOUSING OF THE MAIN GUIDANCE SECTION OF A GUIDED MISSILE

As a result of these studies it was recommended that a magnesium alloy with high damping properties (K1X1) be used for the housing of the main guidance section of the missile and accordingly reduce the resonance amplitudes and the corresponding accelerations occurring in the housing. The main purpose of this part of the investigation on the damping properties of the K1X1 alloy was the determination of the degree of reduction in resonance amplitudes obtained by the use of the K1X1 alloy instead of a conventional magnesium alloy such as the AZ81A for this casting. The reduction of the resonance amplitudes has been determined analytically. The results have been compared with actual test results obtained by vibrating K1X1 and AZ81A alloy castings on a vibration table.

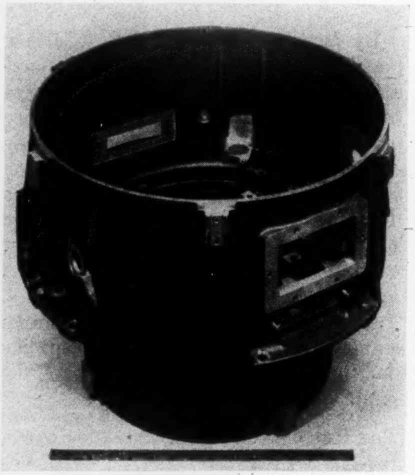


FIG. 12.—Housing for Main Guidance Section of Nike Hercules Missile.

Figure 12 shows the machined casting and Fig. 13 shows the casting mounted on the vibration table. Accelerometers have been mounted at various locations on the casting. The casting has been subjected to a known acceleration at varying frequencies and the corresponding acceleration has been measured by means of accelerometers and recorded on a Visicorder type oscillograph.

A comparison between the K1X1 and the AZ81A alloy castings has been made at the circumference of the upper drum of the casting (point *A* in Fig. 12). Reso-

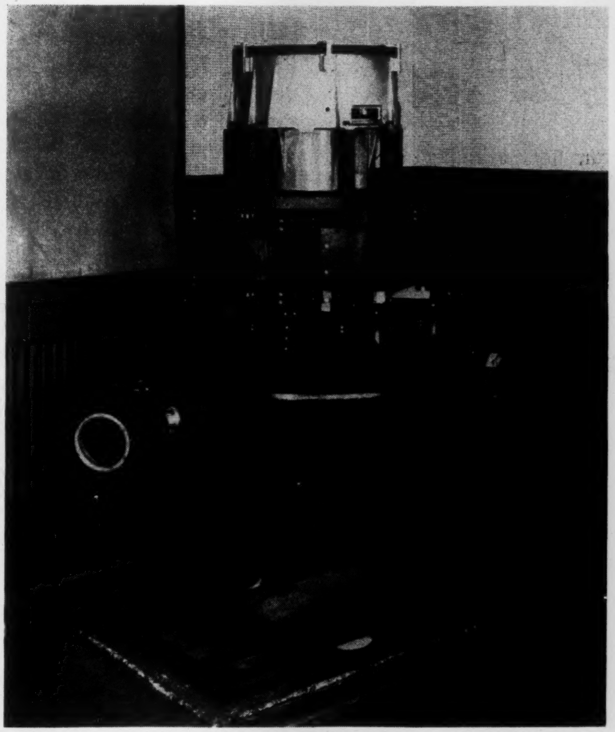


FIG. 13.—Housing for Main Guidance Section Mounted on the Vibration Table.

nance occurred at about 600 cps. Table IV shows the measured accelerations of the drum at resonance, the acceleration of the casting, and the resonance frequency of the drum. Due to limitations of the equipment it was not possible to measure accelerations of the AZ81A alloy casting beyond 1 *g*. At 1 *g* the ratio of the resonance amplitudes of the castings is:

$$R = \frac{18.6}{78.5} 100 = 23.7 \text{ per cent}$$

An attempt has been made to predict the ratio *R* analytically from the known specific damping capacities of the two alloys. For ease of calculation, it has been assumed that the strain distribution along the circumference of the drum is

TABLE IV.—MEASURED ACCELERATIONS ON GUIDANCE HOUSINGS.

Material of Casting	Acceleration of Casting, *g*	Resonance Frequency of Drum, cps	Acceleration of Drum at Resonance, *g*
K1X1 alloy.........	1	615	18.6
K1X1 alloy.........	3	610	55.5
K1X1 alloy.........	5	600	90.0
K1X1 alloy.........	8	590	140.0
AZ81A alloy........	1	610	78.5

parabolic, of the form:

$$\epsilon = \epsilon_m \left[1 - \left(\frac{x}{l}\right)^2\right] \frac{2z}{h} \ldots\ldots (12)$$

where:

ϵ = strain,
ϵ_m = maximum fiber strain,
l = one eighth of circumference of drum,
h = thickness of drum,
z = distance from neutral fiber, and
x = distance from point of maximum fiber strain.

Following a procedure similar to that used for the cantilever beam (see appendix), the following resonance amplification factor is obtained:

$$A_r = \frac{5l^2}{24hx_0} \left[\frac{48\pi h x_0}{5l^2 A_n \alpha_n}\right]^{1/(1+\beta)} \ldots (13)$$

where:

$A_n = A_1 = 0.735$ for K1X1 alloy, and
$A_n = A_2 = 0.564$ for AZ81A alloy.

It is understood, that the moment distribution at resonance is not truly parabolic. However, it is felt that a parabolic moment distribution is a reasonable approximation for the determination of the relative damping capacity of the two alloys proposed for use in the manufacture of the casting. The ratio R becomes:

$$R = 2.32 \frac{\sqrt[6]{hx_0}}{\sqrt[3]{l}}$$

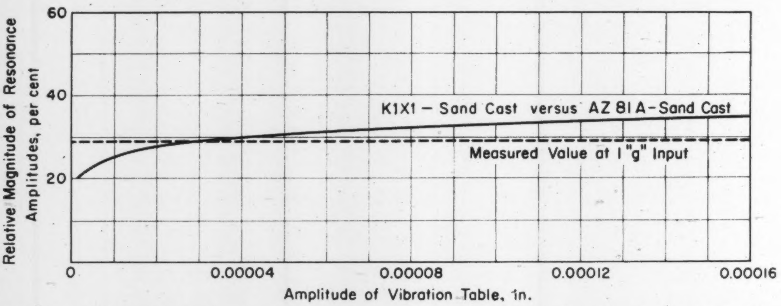


FIG. 14.—Relative Magnitude of Resonance Amplitudes of Housing for the Main Guidance Section of Nike Hercules Missile—Magnesium K1X1 *versus* Magnesium AZ81A Alloys.

This ratio has been plotted in Fig. 14 as a function of x_0 with $l = 10$ in. and $h = 0.25$ in. It is shown here that the ratio does not change substantially within the range of the amplitudes of the vibration table. The agreement between the experimentally determined values and the calculated values is considered very good.

SUMMARY AND CONCLUSIONS

It is possible to calculate the specific damping capacity of a material from simple vibration tests of cantilever beams. From these data the resonance amplitudes of any simple structure can be predicted analytically. In the case of complicated structures the relative damping performance of two different alloys can be determined analytically.

The high-damping magnesium alloy used for this investigation is designated as K1X1. It contains nominally 0.6 to 0.7 per cent zirconium (balance magnesium). The damping properties are not affected by machining, aging or finishing. Heat treatments do not affect the unmachined alloy; however, a reduction in damping has been observed after heat treatment of machined samples. The sand-cast alloy shows better damping properties than the die-cast material. The resonance amplitudes of sand castings produced in the K1X1 alloy show a reduction to approximately 25 to 40 per cent of those occurring in the AZ81A alloy castings, depending on the existing strain distribution.

As a result of this study, production of the main housing, instrument mounting plate and gyro support brackets for the guidance unit is now under way, employing the sand-cast K1X1 alloy. It is also proposed to produce the instrument mounting plate as a die casting. Completely satisfactory sand and die castings in the K1X1 alloy have been produced.

Acknowledgment:

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APPENDIX

Mathematical Relationship Between Damping and Strain Distribution:

The specific damping capacity or the damping capacity of a volume element is:

$$\psi = \frac{\Delta U}{U} = \alpha \epsilon^{\beta} \qquad (14)$$

where:

ψ = specific damping capacity,

ΔU = specific damping energy,

$U = \frac{1}{2}E\epsilon^2$ = specific elastic energy,

ϵ = strain, and

α, β = material constants.

Equation 14 is related to a volume element.

The damping energy and the elastic energy of a structural part is related to damping energy of a volume element by

$$\Delta\bar{U} = \int_{(V)} \Delta U \, dV = \int_{(V)} \psi U \, dV$$

$$= \frac{1}{2} \alpha E \int_{(V)} \epsilon^{2+\beta} \, dV \,.. (15)$$

$$\bar{U} = \int_{(V)} U \, dV = \frac{1}{2} E \int_{(V)} \epsilon^2 \, dV \,.... (16)$$

From Eqs 15 and 16 follows the damping capacity of the part

$$\bar{\psi} = \frac{\Delta\bar{U}}{\bar{U}} = \frac{\int_{(V)} \psi U \, dV}{\int_{(V)} U \, dV} = \alpha \frac{\int_{(V)} \epsilon^{2+\beta} \, dV}{\int_{(V)} \epsilon^2 \, dV} \quad (17)$$

Damping Capacity of a Cantilever Beam:

The strain distribution of a cantilever beam with a rectangular cross-sectional area and a single force operating at its free end is:

$$\epsilon = \epsilon_m \frac{x}{l} \frac{2z}{h} \,........... (18)$$

where:

x = distance from free end,

z = distance from neutral fiber,

l = length of beam,

h = height of beam,

ϵ_m = maximum strain at fixed end of beam, and

ϵ = strain as a function of x and z.

Substituting Eq 18 into Eq 17

$$\bar{\psi} = \alpha \left(\frac{2\epsilon_m}{lh} \right)^\beta \frac{\int_0^b \int_0^{h/2} (xz)^{2+\beta} \, dx \, dz}{\int_0^l \int_0^{h/2} (xz)^2 \, dx \, dz}$$

$$= \alpha \left(\frac{3}{3 + \beta} \right)^2 \epsilon_m{}^\beta \,.................. (19)$$

which gives the relationship between the damping capacity of a unit volume and of the cantilever beam

$$\bar{\psi} = \alpha \left(\frac{3}{3 + \beta} \right)^2 \psi = \bar{\alpha} \epsilon_m{}^{\bar{\beta}} \,.... (02)$$

from which follows

$$\beta = \bar{\beta} \quad \text{and} \quad \bar{\alpha} = \alpha \left(\frac{3}{3 + \beta} \right)^2 \,.. (21)$$

Resonance Amplification Factor of a Cantilever Beam:

The resonance amplification factor of a one degree of freedom vibrator is

$$A_r = \frac{A_m}{x_0} = \frac{2\pi}{\bar{\psi}} \,......... (22)$$

where:

A_r = resonance amplification factor,

A_m = amplitude of mass of vibrating system,

x_0 = amplitude of base of vibrating system, and

$\bar{\psi}$ = damping capacity of vibrating system.

The amplitude of the mass A_m and the damping capacity of the vibrating system $\bar{\psi}$ are functions of the strain. For the cantilever beam

$$A_m = \frac{2l^2 \epsilon_m}{3h} \,........... (23)$$

$$\bar{\psi} = \alpha \left(\frac{3}{3 + \beta} \right)^2 \epsilon_m{}^\beta \,....... (24)$$

Substituting Eqs 23 and 24 into Eq 22 and solving for ϵ_m

$$\epsilon_m = \left[\frac{\pi}{3} \frac{(3 + \beta)^2}{\alpha} \frac{h}{l^2} x_0 \right]^{1/(1+\beta)} \,... (25)$$

By means of Eqs 22, 23, and 24, the resonance amplification factor becomes:

$$A_r = \frac{2}{3} \left[\frac{\pi}{3} \frac{(3 + \beta)^2}{\alpha} \left\{ \frac{l^2}{hx_0} \right\}^\beta \right]^{1/(1+\beta)} \,.. (26)$$

DISCUSSION

MR. J. G. KAUFMAN[1] (*presented in written form*).—The authors have, with their excellent paper, given an immeasurable amount of confidence to those of us who are using a simple system for determining the relative damping properties of metals. We at the Alcoa Research Laboratories have been evaluating the damping properties of aluminum and magnesium alloys using a system very similar to the cantilever beam setup described by the authors. The results of the Alcoa tests have been evaluated in terms of log decrement or specific damping capacity only. It is certainly gratifying to learn of an example where such results were not only accurate barometers of damping behavior but also were used to predict the vibration characteristics of a structural unit such as the Nike-Hercules missile housing.

The only significant difference between the two test setups involves the weight fastened to the end of the specimen during vibration. Other minor differences include the fact that our rectangular specimen has $\frac{1}{4}$ by 2-in. cross-section rather than the $\frac{1}{4}$ by $1\frac{1}{2}$-in. mentioned by the authors and our wave traces are recorded on a Consolidated oscillograph rather than an oscilloscope.

I would like to question the authors regarding the fixed weight. Was the weight used for any specific purpose, for example, uniformity of results? We do not use any weight on the ends of the specimens, and they vibrate only under their own weight. We understand from the work of Person and Lazan (**1**)[2] that this should not affect the results. However, it does seem possible that the data obtained could be more uniform and less subject to secondary vibrations as a result of the added mass.

We would also like to ask the authors whether they have done any work on magnesium alloys containing rare earths, EZ33A (AMA 131), for example. Alcoa tests, both of the type described here and using tuning forks, have indicated that there is considerable potential in these alloys for high damping applications.

MR. H. G. WARRINGTON.[3]—The authors mention that alloys up to 1 per cent zirconium were used. Purely from the practical point of view it would be interesting to know how more zirconium was obtained in the alloy than is in solution at equilibrium in the molten condition.

The effect of thermal conductivity in fundamental work on alloys of this type is of interest. At first glance one would say from the very fact that the

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[2] The boldface numbers in parentheses refer to the list of references appended to this discussion, see p. 892.

[3] Manager, Sales and Technical, Dominion Magnesium, Ltd., Toronto, Ont., Canada.

K1 alloy and the pure magnesium offer a much higher thermal conductivity than AZ80 type of alloy, that thermal conductivity is a good thing, since it assists in dissipating heat. But looking at the slope of the curves, the opposite suggestion comes to mind, that if one could obtain pure magnesium or alloys of the K1 type with every other factor constant, there is the possibility that a lower conductivity might be of assistance from the point of view that the development of high temperatures in extremely local conditions would reduce the modulus locally and increase the opportunities of slip and displacement in the grain itself. I wonder if this question has arisen in taking a more fundamental look in this type of alloy development.

TABLE V.—ROOM TEMPERATURE TENSILE PROPERTIES OF BARS SECTIONED FROM A K1X1-F SLOPE CASTING.

Bar Location	Section Size, in.	Tensile Properties[a]		
		Per Cent Elongation, in 2 in.	Tensile Yield Strength, psi[b]	Tensile Strength, psi
A	5	15.5	59 00	22 800
B	5	17.0	58 00	22 900
C	5	14.5	62 00	22 800
D	2	18.5	61 00	22 800
E	1½	18.5	61 00	22 900
F	1	19.0	62 00	23 100
G	½	21.5	68 00	23 300
H	⅜	23.5	71 00	23 700
I	5/16	26.0	75 00	24 300
J[c]	¼	15.5	75 00	24 000
Average		19.0	65 00	23 300

[a] Results obtained on one bar per location.

[b] 0.2 per cent offset.

[c] Fractured bar showed an oxide inclusion at its periphery.

Separately-cast test bars from the same batch of metal tested at 20 per cent elongation, 8 ksi yield strength and 25 ksi tensile strength.

MR. G. F. WEISSMANN (*author*).—I cannot say. We did not go into that problem.

CHAIRMAN I. V. WILLIAMS.[4]—I think you did look into this question of the effect of higher zirconium.

MR. WEISSMANN.—Yes, but apparently it did not affect the damping very much. It just reduced the mechanical properties of the material. A value of 0.6 per cent appears to give the optimum in damping and mechanical properties.

CHAIRMAN WILLIAMS.—Apparently additional zirconium did not do much good. Only 0.6 per cent goes into solution anyway, so you are just wasting your zirconium.

MR. FRANCIS T. BARATTA.[5]—I was wondering about the comparison between the forced vibration of the specimen and the actual forced vibration of the prototype.

MR. WEISSMANN.—The comparison between the forced vibration of the specimen and the actual forced vibration of the prototype is outlined in the paper for one particular point at the prototype. Similar comparisons have been made at other locations. It has been shown that by means of a known specific damping capacity of a material it is possible to determine the resonance amplification of a steady vibration, provided the strain distribution is known. The real difficulty lies in the determination of the strain distribution. Reasonable assumptions are often necessary to overcome the mathematical difficulties.

MR. K. E. NELSON[6] (*by letter*).—The Bell Telephone Laboratories should be given due credit for expediting the application of these light-weight, high-damping-capacity alloys and for implementing and conducting this test program which ultimately led to the formal introduction of magnesium alloy

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[4] Bell Telephone Laboratories, Murray Hill, N. J.

[5] Applied Physics Branch, Watertown Arsenal Laboratories, Watertown, Mass.

[6] Metallurgical Development, Metallurgical Laboratory, The Dow Chemical Co., Midland, Mich.

K1X1-F. This alloy is currently available on an experimental basis both as die castings and as sand castings, for general customer evaluation.

I am taking this opportunity of offering this discussion as a supplement to the work of the authors.

Castability:

The authors have commented on the relative castability of K1X1 alloy. As a further comment on this subject, it can be pointed out that pattern shrinkage values of about 140 per cent of those used for AZ92A alloy can be used for K1X1 sand castings.

Mechanical and Physical Properties:

Room temperature tensile properties as a function of casting section size were determined using a special laboratory test casting (2). Results are given in the accompanying Table V.

Table VI presents room temperature tensile strength data obtained on separately-cast test bars poured along with castings in the production foundry. Elevated temperature strengths obtained on cast test bars poured in the laboratory as well as bearing and shear strengths and various physical properties are also presented in this table.

TABLE VI.—MECHANICAL AND PHYSICAL PROPERTIES OF SAND-CAST K1X1-F ALLOY.

	Tensile Properties														
	75 F[a]			75 F[b]			200 F[b]			400 F[b]			600 F[b]		
	Per Cent Elongation	Tensile Yield Strength, psi	Tensile Strength, psi	Per Cent Elongation	Tensile Yield Strength, psi	Tensile Strength, psi	Per Cent Elongation	Tensile Yield Strength, psi	Tensile Strength, psi	Per Cent Elongation	Tensile Yield Strength, psi	Tensile Strength, psi	Per Cent Elongation	Tensile Yield Strength, psi	Tensile Strength, psi
Average......	19	7000	25 000	18	8000	24 000	30	7000	17 000	71	5000	8000	78	2000	4000
Minimum.....	11	6000	24 000	..	..	..	..	..	..	..	..	..	..	..	..

Bearing Strength, ksi[c]		Shear Strength, ksi			Density, g per cu cm	Electrical Resistivity, microhm-cm, 68 F	Thermal Conductivity, Btu cm per hr sq cm deg Cent (68 F)	Corrosion Rate,[d] mg per sq cm per day
		Pin Diameter, in.						
Bearing Yield	Bearing Ultimate	⅛	3/16	¼				
17.5	45.9	8.1	7.8	7.4	1.752	5.65×10^{-6}	4.15	0.31

[a] Results on 150 melts, 2 bars per melt using production practices.

[b] Results on 2 bars per test. Melt prepared in the laboratory.

[c] 2d edge distance—8d width. 3/16 in. diameter pin.

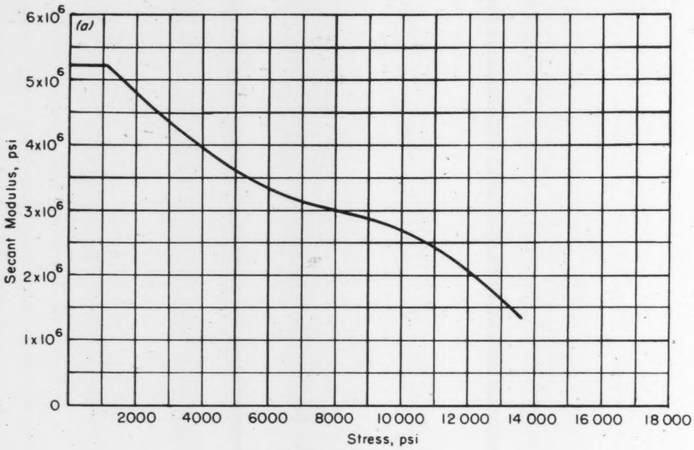
[d] Limited date determined in 3 per cent NaCl solution using the 14-day alternate immersion test described earlier (**5**).

NOTE: Testing procedures for bearing and shear strength determinations are described in other publications (**3, 4**).

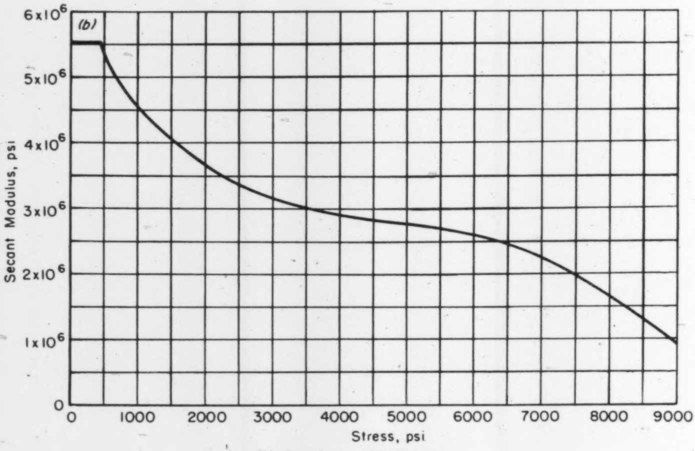
Secant modulus values for die-cast and sand-cast K1X1-F are plotted in Figs. 15(*a*) and (*b*) respectively. The elastic portion of the secant modulus data was obtained by Fenn (3).

Weldability:

Welding studies to date indicate that sand cast K1X1 can be readily repaired by welding. Welding efficiencies on



(*a*) Die-cast K1K1-F.



(*b*) Sand-cast K1K1-F.

FIG. 15.—Secant Modulus *versus* Stress (78 F).

sand-cast K1X1 using EZ33A welding rod were found to be 100 per cent, while K1X1 welding rod gave 80 to 90 per cent efficiencies. Factors such as the effect of welding rod composition on the damping characteristics of K1X1 have yet to be determined.

Protective Coatings:

K1X1-F can be successfully treated chemically with most of the standard treatments used on magnesium alloys. It is not effectively coated by the Dow No. 7 process. When properly chemically treated and painted, this alloy should perform satisfactorily in normal service.

MR. JEROME E. RUZICKA[7] (*by letter*).—I would like to congratulate the authors on their fine presentation of the information developed in their studies. However, I feel it should be pointed out that no data have been presented on the resonance amplification factor for displacement inputs of less than 0.001 in. The variation of the resonance amplification factor with input displacement is shown in Fig. 11(*a*) for input displacements greater than 0.001 in. For the practical situation where the input motion exciting a substructure of a mechanical system is quite small—perhaps in the order of magnitude of 0.0001 in.—the strain levels developed in the structure are very low. Since the damping capacity decreases for decrease in strain levels, the resonance amplification must necessarily increase.

Generally speaking, the resonance amplification factor takes a very sharp rise when the input motions become very small. One might be misled by the fact that the authors chose to use rectangular coordinates in Fig. 11(*a*). By viewing the slope of the curves at say 0.001 in., it appears that reasonably low values of the amplification factor could be expected at the lower input levels. I suspect that the amplification factor would be very much higher than that which would be obtained by projection of the curves into the region of low input excitation. This difficulty could have been avoided by use of a logarithmic scale on the amplitude of vibration table.

From a practical viewpoint, the authors have not given sufficient proof that the new alloy is a vast improvement over existing alloys for use in the design of structures which must operate in a steady-state vibration environment. In order to answer the question as to whether or not the new alloy is superior to existing alloys, it must be proven that the resonance amplification factor is satisfactorily reduced for extremely low displacement input conditions.

MR. G. F. WEISSMANN (*author's closure*).—The authors wish to thank Mr. Nelson for his discussion of this paper. The remarks on castability, mechanical and physical properties, weldability and protective coatings are valuable supplements to this paper.

In reply to Mr. Kaufman's remarks regarding the purpose of the weight at the end of the test specimens, the author wants to state that this weight was attached to obtain a simple strain distribution in the cantilever beam. As has been explained in this paper and others (reference (3) and (4) of paper), the damping capacity is often affected by the strain distribution. Hence, it cannot be expected that test specimens with and without a weight at the free end will render identical results. Person and Lazan (1) state only that the effect of a static mean bending stress on the damping associated with a given alternating bending stress is small for material with little magnetomechanical effect. The stress distribution remained essentially the same in all their experiments.

Regarding the question on magnesium alloys containing rare earth, the authors have not done enough work to judge their value for possible damping applications.

Mr. Ruzicka appears to be very concerned with the resonance amplification factor for displacement inputs of less than 0.001 in. He states very correctly that the damping capacity decreases with a decrease in the strain level and that the resonance amplification factor would therefore increase accordingly. The use of logarithmic coordinates for both the resonance amplification factor and the amplitudes of the vibration table would perhaps show better the superior damping capabilities of the K1X1 alloy compared to the conventional AZ81A alloy especially at low strain values. As far as practical applications are concerned, the authors believe it has been shown that the use of this alloy reduces the resonance amplifications substantially for the particular structure considered here. Figure 14 of the paper shows that by use of this alloy the resonance amplitudes at one particular location have been reduced to about one fourth of the conventional alloy at table amplitudes of 0.0001 in. Similar results have been obtained at other locations. This appears to be sufficient proof of the higher damping capabilities especially at low strain values.

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[7] Barry Controls, Inc., Watertown, Mass.

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A STUDY OF THE VARIABILITY IN THE MECHANICAL PROPERTIES OF ALLOY A PHOSPHOR BRONZE STRIP*

By M. N. Torrey,[1] G. R. Gohn,[1] and M. B. Wilk[1]

Synopsis

This paper describes the use of an incomplete Latin square design for studying the variability in the physical and mechanical properties of grade A phosphor bronze strip. The experiment was designed so that the over-all variability can be partitioned into (1) variability among columns (along the length of the strip), (2) variability among rows (across the width of the strip), (3) variability among milling lots, and (4) residual variability. The actual design used is given together with the method of analyzing the observed data and the test results obtained.

A by-product of the experiment is a comparison of different characteristics in the same group of specimens with respect to variability, for example, proportional limit values showed greater residual variability than did the tensile or 0.2 per cent offset yield strength values.

Two recent ASTM symposia (**1**,**2**)[2] and numerous individual papers have indicated that fatigue is a statistical phenomenon which requires further study. Recently, Torrey and Gohn (**3**) presented results from a comprehensive statistical study of fatigue, which indicated the need for additional studies of this type as well as a need for some knowledge as to the variability in other mechanical properties. A statistically designed experiment was therefore undertaken to determine how mechanical properties, such as tensile strength, elastic properties, and hardness, varied along the length and width of a strip of phosphor bronze; to study the effects of procedures associated with their measurement; and to check on possible relationships among the various properties. The present paper summarizes some of the results of this investigation.

Material Investigated

The material investigated was a strip of alloy A phosphor bronze having a nominal thickness of 0.040 in. and a nominal composition of 5 per cent tin, 0.05 per cent phosphorus, and the balance copper. It was manufactured by the Riverside Metal Co., Division of H. K. Porter Co., Inc., of Riverside, N. J. from a 9 by $2\frac{1}{2}$ by 36-in. ingot cast from

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* Presented at the Sixty-first Annual Meeting of the Society, June 22–27, 1958 and re-presented at the Fall Meeting of Committee B5 at Lake Placid on September 15, 1958.

[1] Technical Staff, Bell Telephone Laboratories, Inc., New York, N. Y.

[2] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 910.

a charge made from electrolytic copper, Pass No. 1 tin, and 15 per cent phosphor copper. These materials were melted in a low-frequency induction furnace and cast in a copper, water-cooled mold.

The $2\frac{1}{2}$-in. thick ingot was cold rolled to 1.250 in. and annealed at 1250 F; cold rolled to 0.625 in., annealed at 1250 F, and scalped. The material was then cold rolled to 0.250 in., annealed at 1100 F, cold rolled to 0.064 in., and annealed at 900 F to a ready-to-finish grain size of 0.015 mm, after which it was cold rolled to the final thickness of 0.040 in.

One hundred feet of this material, supplied in 17 lengths each approximately 6 ft long and 7 in. wide, was used in these tests. The 7 in. wide lengths were obtained from the 9-in. strip by slitting, during which operation the outside inch was removed from both edges of the strip. For purposes of identification each of the 17 lengths was numbered consecutively from 01 to 17, the number having been stamped on the entering end of each strip by the manufacturer as shown in Fig. 1.

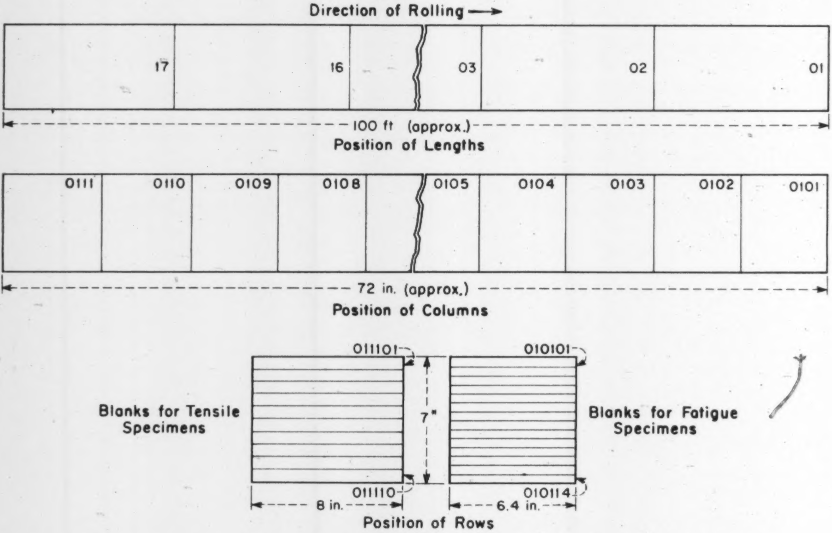
Direction of Rolling
17
16
03
02
01
100 ft (approx.)
Position of Lengths
0111
0110
0109
0108
0105
0104
0103
0102
0101
72 in. (approx.)
Position of Columns
011101
010101
Blanks for Tensile Specimens
7"
Blanks for Fatigue Specimens
011110
010114
8 in.
6.4 in.
Position of Rows

FIG. 1.—Location of Lengths, Columns, and Rows in Phosphor Bronze Strip.

IDENTIFICATION OF SPECIMENS

Each of the 17 lengths described in the preceding paragraph was divided into 11 columns; two additional digits, 01 to 11, identify the location of each column in each length. Ten lengths were chosen at random[3] from the 17 lengths, and one column was chosen at random within each of these ten lengths before the material was laid out. The columns so chosen were 8 in. in length; each of the remaining columns was approximately 6.4 in. long, the length required for fatigue test specimens. The 8-in. columns were divided into 10 rows which were identified by the numbers 01 to 10. Thus by the use of six digits each specimen can be identified as to the length, column, and row from which it was taken. For example, a specimen numbered 011110 was taken from the tenth row in the eleventh column of the first length (Fig. 1).

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[3] Random number tables are included in many statistical texts, for example, references (**4**) and (**5**).

After the entire lot of material was laid out and numbered as described, the individual rectangular blanks were sheared from the several lengths, coated with lard oil to prevent corrosion, and stored in sealed test tubes until required for test.

TABLE I.—INCOMPLETE LATIN SQUARE DESIGN.

Note.—*D* and *F* each designate separate milling lots of 10 specimens. $(B + E)$ and $(A + C)$ designate two separate milling lots, each of 20 specimens.

Column	1	2	3	4	5	6	7	8	9	10
Designation	0111	0402	0506	0703	1006	1103	1201	1302	1410	1709
Row:										
01	...	A	B	...	C	D	...	...	E	F
02	C	...	D	...	E	F	...	A	...	B
03	...	F	...	D	A	...	C	E	B	...
04	F	D	C	B	...	A	E	...	...	...
05	D	...	E	F	...	B	...	...	A	C
06	B	C	F	A	...	...	D	...	...	E
07	A	...	...	E	D	C	...	B	F	...
08	...	E	...	C	B	...	A	F	...	D
09	E	B	A	...	...	...	F	D	C	...
10	...	...	...	...	F	E	B	C	D	A

Properties Investigated

The properties investigated in this study included the mechanical properties—modulus of elasticity, proportional limit, yield strengths at 0.01 and 0.2 per cent offset, tensile strength, elongation (per cent in 2 in.), and Rockwell hardness numbers, B and 30T scales—as well as thickness, width, and area. Of these properties, only the thickness, width, and two Rockwell hardness values were measured directly. The others were calculated from observed values of load, width, thickness, and length by means of well-established relationships.

Experimental Design

The natural sources of variability for this experiment were along the length and across the width of the strip of metal and differences among the milling lots in pretest specimen preparation. The first two can be summarized according to columns and rows, respectively, as described previously. Because a stack of specimens to be milled in one operation could not exceed 1.0 in. in thickness, a single milling lot could not contain more than 23 specimens of 0.040-in. material plus the two outside blanks.

Since the width of the strip allowed ten rows of specimens for tension tests, selecting ten columns from the available 187 gave rise to 100 potential mechanical test specimens, each classified by row and by column.

A class of experimental designs known as Latin square designs is often especially appropriate where the material may be naturally cross-classified according to two criteria—rows and columns. These designs are based on a geometric configuration, the Latin square. A $k \times k$ Latin square is an arrangement of k distinct objects (Latin letters) in k rows and k columns such that every distinct letter appears once in every row and

TABLE II.—MECHANICAL PROPERTIES OF HARD TEMPER ALLOY A (5 PER CENT TIN) PHOSPHOR BRONZE STRIP 0.015 MM READY-TO-FINISH GRAIN SIZE.

Serial No. of Specimen	Order of Test	Thickness, in.	Width, in.	Area, sq in.	Modulus of Elasticity, psi	Proportional Limit, psi	Yield Strength, psi		Tensile Strength, psi	Elongation in 2 in., per cent	Rockwell Hardness	
							0.01 per cent Offset	0.2 per cent Offset			B Scale	30T Scale
Milling lot D:												
110301	13	0.0406	0.4980	0.020219	17.10 × 10^6	52 400	61 300	84 800	89 300	10.0	92.5 to 92.7	77.4 to 78.0
050602	29	0.0405	0.5003	0.020262	16.92	53 300	62 200	84 900	89 500	8.5	92.2 to 92.2	77.7 to 77.8
070303	26	0.0416	0.4983	0.020729	16.54	51 600	61 700	84 400	88 800	10.0	92.3 to 92.2	78.1 to 76.5
040204	21	0.0416	0.4993	0.020771	16.75	53 900	62 600	84 700	89 200	11.5	92.3 to 92.1	78.7 to 78.9
011105	33	0.0412	0.4998	0.020592	16.75	53 400	61 200	83 800	89 200	10.5	92.1 to 91.8	78.5 to 78.4
120106	46	0.0409	0.4996	0.020434	16.92	53 300	61 700	83 700	88 000	9.0	91.6 to 91.9	78.8 to 78.0
100607	37	0.0413	0.4994	0.020625	16.86	52 400	60 600	83 200	88 000	10.5	91.8 to 92.1	77.8 to 77.8
170908	15	0.0409	0.4984	0.020385	16.72	54 000	63 800	84 900	89 300	9.0	92.3 to 92.3	77.4 to 79.0
130209	07	0.0407	0.5002	0.020358	16.51	52 600	61 900	83 800	88 800	12.0	92.3 to 92.3	77.9 to 78.3
141010	09	0.0390	0.4996	0.019484	17.35	55 400	64 700	86 500	88 400	7.0	92.0 to 92.2	78.5 to 78.3
Milling lot F:												
170901	36	0.0405	0.4976	0.020153	16.63	52 600	62 500	83 600	88 100	8.5	92.0 to 91.9	78.0 to 79.2
110302	43	0.0411	0.4954	0.020361	16.65	52 100	60 900	84 200	89 400	10.5	92.4 to 92.9	78.0 to 79.2
040203	38	0.0415	0.4997	0.020738	16.77	52 600	61 700	84 100	89 500	11.0	92.6 to 92.6	78.0 to 78.0
011104	59	0.0412	0.4998	0.020592	16.75	52 400	62 200	84 500	88 800	11.0	92.4 to 92.4	78.1 to 77.2
070305	44	0.0416	0.4982	0.020725	16.93	53 100	60 800	83 000	88 500	11.0	91.8 to 91.7	77.8 to 77.9
050606	11	0.0409	0.4992	0.020417	17.09	53 900	62 700	83 800	87 900	8.5	91.8 to 91.7	77.8 to 78.3
141007	08	0.0400	0.4973	0.019892	17.04	55 300	63 800	85 500	89 500	7.0	91.7 to 91.4	78.5 to 78.8
130208	06	0.0409	0.4997	0.020438	16.73	51 900	61 700	83 900	88 300	11.0	92.3 to 92.3	78.8 to 77.9
120109	05	0.0403	0.4990	0.020110	16.76	54 200	62 700	84 300	88 300	9.0	92.2 to 92.7	79.2 to 78.6
100610	42	0.0404	0.4977	0.020107	16.72	52 200	61 700	83 800	88 600	8.5	92.4 to 92.2	78.8 to 79.3
Milling lot B:												
050601	49	0.0402	0.4933	0.019831	16.72	53 500	62 000	83 700	87 100	8.5	92.3 to 92.1	78.3 to 79.1
170902	60	0.0409	0.4970	0.020327	16.77	52 600	63 000	83 900	88 900	8.0	92.7 to 92.4	79.2 to 79.0
141003	41	0.0398	0.4962	0.019749	17.12	53 700	61 800	86 300	89 500	8.0	92.5 to 92.5	78.2 to 78.5
070304	20	0.0417	0.4903	0.020446	16.58	53 800	64 100	84 900	88 700	9.5	92.8 to 92.2	77.8 to 79.5
110305	27	0.0413	0.4978	0.020559	16.92	52 000	61 300	83 700	88 600	10.5	92.2 to 92.1	76.8 to 77.3
011106	53	0.0412	0.4987	0.020546	16.50	54 000	61 300	83 000	88 100	9.5	92.1 to 92.1	78.1 to 77.6
130207	17	0.0411	0.4962	0.020394	16.62	53 400	61 800	84 300	87 900	8.5	92.0 to 92.1	78.6 to 76.2
100608	50	0.0411	0.4959	0.020381	16.73	52 500	61 800	84 100	88 400	10.5	92.2 to 92.2	79.2 to 79.2
040209	02	0.0410	0.4980	0.020418	16.79	54 400	62 700	84 000	88 200	10.0	92.3 to 92.4	79.1 to 79.0
120110	35	0.0395	0.4978	0.019663	16.67	52 900	62 000	85 400	90 200	8.0	92.3 to 92.3	79.0 to 78.9

Milling lot E:											
141001	32	0.0392	0.4979	0.019518	17.22 × 10^6	56 400	64 600	86 800	88 400	6.5	92.1 to 92.4	79.5 to 78.4
100602	04	0.0411	0.4971	0.020431	16.78	51 900	61 700	83 900	88 200	9.5	92.2 to 92.2	77.6 to 78.4
130203	48	0.0412	0.4982	0.020526	16.66	52 600	60 400	83 800	89 600	9.0	92.3 to 92.3	78.9 to 78.5
120104	56	0.0409	0.4969	0.020323	16.73	52 200	62 000	84 600	89 700	9.0	92.3 to 92.3	79.0 to 79.2
050605	47	0.0409	0.4984	0.020385	16.72	53 500	61 800	83 600	88 900	8.5	92.2 to 92.2	77.8 to 78.0
170906	39	0.0411	0.4960	0.020386	16.82	52 500	60 800	83 400	88 100	8.5	92.1 to 92.0	77.8 to 77.5
070307	52	0.0415	0.4959	0.020580	16.66	53 500	62 200	83 800	89 000	9.5	92.0 to 92.1	78.1 to 78.8
040208	45	0.0413	0.4981	0.020572	16.71	53 000	61 200	83 600	89 200	11.0	92.2 to 92.3	77.8 to 77.7
011109	30	0.0409	0.4934	0.020180	16.80	52 500	61 000	83 700	88 300	11.5	92.2 to 92.2	78.2 to 79.2
110310	22	0.0403	0.4958	0.019981	16.64	54 100	63 100	84 800	89 200	9.0	92.3 to 92.5	79.7 to 79.2
Milling lot A:											
040201	10	0.0411	0.5008	0.020583	16.95	52 500	62 200	83 300	87 500	11.0	92.1 to 92.1	78.4 to 78.0
130202	24	0.0411	0.5001	0.020554	16.45	56 000	63 200	84 200	88 200	9.5	92.5 to 92.3	79.0 to 78.7
100603	28	0.0413	0.4936	0.020386	16.63	52 000	61 800	84 400	89 100	9.0	92.3 to 92.5	77.9 to 78.0
110304	01	0.0413	0.4983	0.020580	16.66	55 400	62 200	84 500	88 800	11.0	92.5 to 92.7	78.8 to 78.2
141005	03	0.0397	0.4989	0.019806	17.51	55 500	62 600	86 300	89 100	8.5	91.9 to 92.1	78.8 to 77.9
070306	19	0.0416	0.4970	0.020675	16.68	54 200	62 400	83 700	87 600	10.0	91.9 to 92.0	79.0 to 78.5
011107	31	0.0411	0.4990	0.020509	16.81	53 100	61 400	83 400	88 800	11.5	92.1 to 92.0	77.3 to 78.0
120108	54	0.0405	0.4983	0.020181	16.99	52 500	61 900	84 700	89 900	8.5	91.8 to 92.3	78.0 to 79.2
050609	25	0.0405	0.4993	0.020222	16.81	52 400	62 300	84 600	89 000	9.0	92.3 to 92.5	78.0 to 78.0
170910	12	0.0401	0.4970	0.019930	16.96	57 700	65 200	85 000	89 200	8.5	92.3 to 92.5	79.2 to 78.9
Milling lot C:											
100601	58	0.0409	0.4972	0.020335	16.48	53 100	62 900	83 800	87 500	9.0	92.0 to 92.1	78.1 to 78.2
011102	51	0.0409	0.4990	0.020409	16.70	53 900	62 700	84 300	89 200	9.0	92.7 to 92.7	78.0 to 78.5
120103	34	0.0409	0.4985	0.020389	16.72	54 900	61 800	84 900	89 300	9.0	92.5 to 92.5	78.2 to 78.9
050604	40	0.0409	0.4993	0.020421	16.79	53 900	62 700	84 700	89 300	8.5	92.5 to 92.5	78.7 to 78.9
170905	16	0.0411	0.4968	0.020418	16.94	53 900	61 700	84 200	89 200	9.0	92.0 to 92.1	78.2 to 78.1
040206	55	0.0416	0.4989	0.020754	16.71	53 000	61 700	82 900	88 700	10.5	91.9 to 92.0	77.8 to 78.2
110307	14	0.0413	0.4967	0.020514	16.71	53 600	62 400	83 800	88 100	10.0	92.0 to 91.8	78.8 to 79.0
070308	23	0.0413	0.4961	0.020489	16.64	54 200	63 000	84 900	89 700	10.0	92.7 to 92.5	78.5 to 78.5
141009	18	0.0392	0.4981	0.019526	17.17	55 800	66 100	87 600	90 500	7.5	91.8 to 92.3	78.9 to 78.2
130210	57	0.0402	0.4981	0.020024	16.65	52 900	60 900	83 400	88 100	11.5	92.2 to 92.1	79.3 to 79.0

every column. An example for $k = 3$, is as follows:

$$\begin{array}{ccc} A & B & C \\ B & C & A \\ C & A & B \end{array}$$

Classically, such designs are of value in comparing k treatments, corresponding to the distinct letters, when major sources of variation in the material may be associated with rows and columns. Comparisons of the treatment effects, which are balanced with respect to the additive contributions of rows and columns, may then be made and the statistical analysis involved is computationally simple.

The basic idea of the Latin square design appeared quite appropriate to the present experimental situation, with milling lots regarded as the treatments. Thus, for example, one might employ 10 milling lots, each of 10 specimens, made up from our 10 × 10 square according to a Latin square design. This would, however, have involved more specimens than desired because general time, labor, and test facility considerations indicated that about 50 or 60 specimens could be used in the experiment. Also, if all milling lots were of equal size the experiment would not have indicated anything about the dependence of variability within milling lots on the lot size.

At the expense of a somewhat more tedious statistical analysis, a modified Latin square design, formally a 10 × 10 Latin square with 4 "missing" treatments, was employed. This reduced the number of specimens required to 60 but still allowed all 10 rows and 10 columns to be represented. The 10 × 10 Latin square configuration was taken from the tables of Fisher and Yates (4), and the rows, columns, and letters of this configuration were reordered at random, retaining the original number designation of the rows and columns, which were identified with the row by column layout of the specimens. Finally, four of the ten letters were randomly chosen and deleted from the design. Each of the six remaining Latin letters then designated 10 specimens. To shed light on the effect of size of milling lot, two pairs of letters were amalgamated. Thus the design called for two milling lots of 20 specimens each and two of 10 specimens each. With arbitrary relabeling of letters, the design shown in Table I resulted.

In this design the letters D and F each designate 10 specimens used in two separate milling lots of 10. Each pair $(B + E)$ and $(A + C)$ designate 20 specimens used in two separate milling lots of 20.

The position of specimens within lots, the order in which the lots were milled, and the order in which the individual specimens were tested were randomized. The general object of randomizing in this experiment, as it is in others, is to keep unknown or uncontrolled effects from being *systematically* confounded with factors under deliberate study.

Table II gives the order of milling, order of specimens in the stack, and order of test. For example, the first group of tension test specimens to be machined was milling lot D, the blanks being stacked in the order given in the first column of the table. The machined specimens were tested in the order given in the second column of Table II.

SPECIMEN PREPARATION AND TEST METHODS

The numbered blanks were stacked in the order given in Table II, as determined by the randomization of the experimental design. They were prepared by the conventional cross-milling technique using a spiral fluted form cutter. The blanks were milled in four lots: Lots D and F were milled separately as lots of 10 while lots $(A + C)$ and $(B + E)$ were milled as lots of 20 each.

After milling, the specimens were measured in the order given in Table II. For each specimen, the point of minimum thickness was located. The thickness and width at that point were measured with a micrometer having a nominal precision of ±0.00005 in. The corresponding area is essentially the minimum cross-sectional area of the specimen.

The Rockwell hardness tests were then made as prescribed in ASTM Methods E 18.[4] A spot anvil and a new $\frac{1}{16}$-in. diameter steel ball were used for each series of tests. Prior to and following completion of the test, the calibration of the hardness tester was checked against a standard block and found to be within the specified limits. The Rockwell hardness numbers were read to ±0.1. One reading was taken at each end of the tensile specimen. The specimens were tested in the order given in the second column of Table II.

The tension tests were made as prescribed in ASTM Methods E 8,[5] using test specimens in accordance with the dimensions shown in Fig. 7 of Methods E 8. A 60,000-lb Baldwin-Tate-Emory testing machine with Templin jaws and a microformer automatic extensometer (P101 M21) was used. Preloads of 20 to 40 lb were applied to each specimen prior to the attachment of the extensometer. During test, the speed of test (rate of jaw separation) was constant for all tests. In the load-strain charts, the magnifications were such that 1 in. = 200 lb load and 0.002 strain. Calibration of the testing machine showed that the load scale used was accurate to ± 0.06 per cent.

Load values were read from the curves on the chart recordings to ± 5 lb for proportional limit and yield strength at 0.01 per cent offset. For yield strengths at 0.2 per cent offset, the load values were read from the curves to ±2.5 lb. These ranges take into account the uncertainty in determining the offset point as well as the uncertainty in determining the load value at that point. The maximum load values for tensile strength were read from the testing machine scale to $\pm\frac{1}{2}$ lb. Strain values were read from the chart to ±0.00002. Elongation values were derived from the measurement of the length of the specimen between gage points before and after fracture; the lengths were read to ±0.005 in. The tension tests were made in the order listed in the second column of Table II.

All tests were made by one operator who also read the values from the load-strain charts.

STATISTICAL ANALYSIS

The purpose of the statistical analysis was to evaluate objectively any systematic effects in the mechanical properties that could be associated specifically with the row, column, or milling lot classifications. The large variabilities inherent in the test material, in methods of specimen preparation, and in some of the test procedures make it especially desirable to employ objective statistical procedures.

The basic preliminary tools for studying the data were a least squares analysis and an analysis of variance. Both these procedures were complicated by the fact that the design used was incomplete; that is, only 60 of the available 100 blanks were tested. Thus the six (pseudo) milling lots were balanced with respect to rows and columns, but the row and column classifications were not balanced with respect to each other (see Table I).

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[4] Tentative Methods of Test for Rockwell Hardness and Rockwell Superficial Hardness of Metallic Materials (E 18 – 57 T), 1958 Book of ASTM Standards, Part 3.

[5] Tentative Methods of Tension Testing of Metallic Materials (E 8 – 57 T), 1958 Book of ASTM Standards, Part 3.

The model used for analysis is indicated by the following:

Observation = (over-all mean) + (row effect) + (column effect) + (milling lot effect) + (residual)

The application of least squares to this model leads to $1 + 10 + 10 + 6 = 27$ equations which define the estimates of the various effects. These equations, which are linear in the estimates of the effects, are not however independent of each other. To obtain unique solutions, it is both customary and reasonable to employ the restrictions:

Sum of estimates of row effects = 0

Sum of estimates of column effects = 0

Sum of estimates of milling lot effects = 0

With such constraints, the effect of row 1, for example, is to be interpreted as the deviation between the average response of specimens in that row from the over-all mean response.

The equation for estimating the over-all mean then becomes:

60 × (estimate of over-all mean) = grand total

The six equations for the (pseudo) milling lot effects each have the form:

10 × (estimate of over-all mean) +
10 × (estimate of milling lot effect) =
total for that milling lot.

The equations for the row effects and those for the column effects, however, have a structure exemplified by the equations associated with the effects for row 1 and column 1 which are as follows (6):

$$6\hat{\mu} + 6\hat{r}_1 + \hat{c}_2 + \hat{c}_3 + \hat{c}_5 + \hat{c}_6 + \hat{c}_9 + \hat{c}_{10} = R_1$$

$$6\hat{\mu} + 6\hat{c}_1 + \hat{r}_2 + \hat{r}_4 + \hat{r}_5 + \hat{r}_6 + \hat{r}_7 + \hat{r}_9 = C_1$$

Since we have imposed the conditions $\sum_{i=1}^{10} r_i = \sum_{j=1}^{10} c_i = 0$, these are equivalent to

$$6\hat{\mu} + 6\hat{r}_1 - \hat{c}_1 - \hat{c}_4 - \hat{c}_7 - \hat{c}_8 = R_1$$

$$6\hat{\mu} + 6\hat{c}_1 - \hat{r}_1 - \hat{r}_3 - \hat{r}_8 - \hat{r}_{10} = C_1$$

where:

$\hat{\mu}$ = estimate of effect due to the over-all mean,
$\hat{r}_1$ = estimate of the effect due to row 1,
$\hat{c}_1$ = estimate of the effect due to column 1,
R_1 = total for row 1,
C_1 = total for column 1, etc.

These two sets, each of ten equations, must be solved simultaneously to evaluate the least squares row and column effect estimates. This may be done iteratively by finding row effects as exemplified by:

$$6\tilde{r}_1 = R_1 - 6\hat{\mu} + \tilde{c}_1 + \tilde{c}_4 + \tilde{c}_7 + \tilde{c}_8$$

using "trial value" $\tilde{c}$ of the column effects. The $\tilde{r}$ values are then used in the column equations as exemplified by

$$6\tilde{c}_1 = C_1 - 6\hat{\mu} + \tilde{r}_1 + \tilde{r}_3 + \tilde{r}_8 + \tilde{r}_{10}$$

Then the new $\tilde{c}$ values are used in equations for $\tilde{r}$ values, etc., until the difference between the latest value and the preceding value is zero to the desired number of places.

Such a process is convergent and practical, even with a desk calculator, if graphical extrapolation is used as a guide.

In the present work, this computation was carried out on an IBM 650 computer. This was especially advantageous here because a parallel analysis was carried out on each of 11 characteristics.

In the analysis of variance of this experiment, the 60 observations on a particular characteristic, such as tensile strength, are partitioned into sets of “degrees of freedom” as follows:

Source	Degrees of Freedom
Over-all mean	1
Rows	9
Columns	9
Milling lots	5
Residual	36

where:

$\hat{m}_k$ = estimate of milling effect for the kth milling lot, and

M_k = total for the kth milling lot.

The analysis of variance table is completed as follows:

Source of Variability	Degrees of Freedom	Sum of Squares	Mean Square
Over-all mean	1	S_1	S_1
Rows and columns	18	S_2	$S_2/18$
Columns ignoring rows	9	S_3	
Rows eliminating columns	9	$(S_2 - S_3)$	$(S_2 - S_3)/9$
Rows ignoring columns	9	S_4	
Columns eliminating rows	9	$(S_2 - S_4)$	$(S_2 - S_4)/9$
Milling lots	5	S_5	$S_5/5$
Residual	36	$S_6 = (\Sigma y_i^2 - S_1 - S_2 - S_5)$	$S_6/36$

Since the design is not completely balanced, the sum of squares for rows and columns cannot be computed from the simple row and column totals. Having in hand, however, the individual observations, the totals for rows and columns, and the least squares estimates of the effects of rows and columns, the various sums of squares may be found as follows:

Sum of squares due to mean,

$$S_1 = 60\hat{\mu}^2 = 60\left(\frac{\Sigma y_i}{60}\right)^2$$

where y_i = an individual observation.

Sum of squares due to rows and columns,

$$S_2 = \Sigma \hat{r}_i R_i + \Sigma \hat{c}_j C_j$$

Sum of squares due to columns ignoring rows,

$$S_3 = \frac{\Sigma C_j^2}{6} - 60\hat{\mu}^2$$

Sum of squares due to rows ignoring columns,

$$S_4 = \frac{\Sigma R_i^2}{6} - 60\hat{\mu}^2$$

Sum of squares due to milling lots,

$$S_5 = \Sigma \hat{m}_k M_k = \frac{\Sigma M_k^2}{10} - 60\hat{\mu}^2 .$$

The variability among rows is then evaluated from the comparison of the mean squares $(S_2 - S_3)/9$ and $S_6/36$. Similarly, for columns the means squares from $(S_2 - S_4)/9$ and $S_6/36$ are compared. The quantity $\sqrt{S_6/36}$ may be regarded as an estimate of the standard deviation of the residual variation after eliminating the effects due to the assignable causes—namely due to rows, columns, and milling lots.

The five degrees of freedom associated with milling lots may be meaningfully partitioned further. It may be recalled that the six “letters” were assigned to two milling lots of 10 (D and F), and two milling lots of 20 ($B + E$ and $A + C$). It is of interest then to make the following breakdown:

Source	Degrees of Freedom	Sum of Squares
Between milling lots of 10 (D *versus* F)	1	S_{51}
Between milling lots of 20 [$(B + E)$ *versus* $(A + C)$]	1	S_{52}
Lots of 10’s *versus* lots of 20’s [$2(D + F)$ *versus* $(B + E) + (A + C)$]	1	S_{53}
10’s within 20’s (B *versus* E)	1	S_{54}
10’s within 20’s (A *versus* C)	1	S_{55}
		S_5

The sum of squares can be obtained as follows:

$$S_{51} = \frac{(\hat{m}_D - \hat{m}_F)^2}{2} \times 10$$

$$S_{52} = \frac{(\hat{m}_B + \hat{m}_E - \hat{m}_A - \hat{m}_C)^2}{4} \times 10$$

$$S_{53} = \frac{(2(\hat{m}_D + \hat{m}_F) - \hat{m}_B - \hat{m}_E - \hat{m}_A - \hat{m}_C)^2}{12} \times 10$$

$$S_{54} = \frac{(\hat{m}_B - \hat{m}_E)^2}{2} \times 10$$

$$S_{55} = \frac{(\hat{m}_A - \hat{m}_C)^2}{2} \times 10$$

Each of the sources of variability may then be evaluated by comparison of its mean square with the residual mean square.

TEST DATA

Table II gives the test data, by milling lots and the order of the specimens within each stack, for thickness, width, area, and the eight mechanical properties determined from the test specimens.

Figure 2 shows the individual observed values of thickness, width, and the eight mechanical properties by order of test. The horizontal lines are the medians of the 60 observations for each characteristic. Long runs of eleven or more points on either side of the median would indicate a change, at the 0.01 significance level, in the testing procedure or in the method of reading the observed values if only one characteristic were considered (7). The longest run observed was eleven points above the median for proportional limit. Since a similar run is not present for any of the related characteristics, it is concluded that no significant changes in the test procedures occurred during the experiment.

STATISTICAL ANALYSIS OF TEST DATA

The data given in Table II were analyzed by the methods outlined previously. Table III summarizes the mean square values obtained for the major sources of variability as well as for the residual, a measure of the variability among test specimens combined with the variability due to test procedures. Table IV gives the ratios of the mean squares to the residual mean square; significantly large values are marked with asterisks and suggest the presence of one or more assignable causes of variation.

Variations in Material:

The most important variation in the material with respect to columns and rows is for thickness. The significant variation of thickness affects the variation of area, and is correlated with the variation in modulus of elasticity, the proportional limit, the yield strengths, and tensile strength (which are based on load divided by area) as well as elongation.

Figure 3 presents graphically, for all characteristics, the estimates of the column and row effects. The deviations from the mean, as shown in Fig. 3, suggest in a general way the relationships among the various properties. The statistical significance of the deviations cannot be judged from the figure but are evaluated objectively through the results of the analysis of variance given in Tables III and IV.

It may be noted that the specimens from column 9 (or 1410) are thinner by more than 0.001 in., on the average, than the specimens from the other columns. The estimates for area and elongation are also low for that column while the estimates of modulus of elasticity, pro-

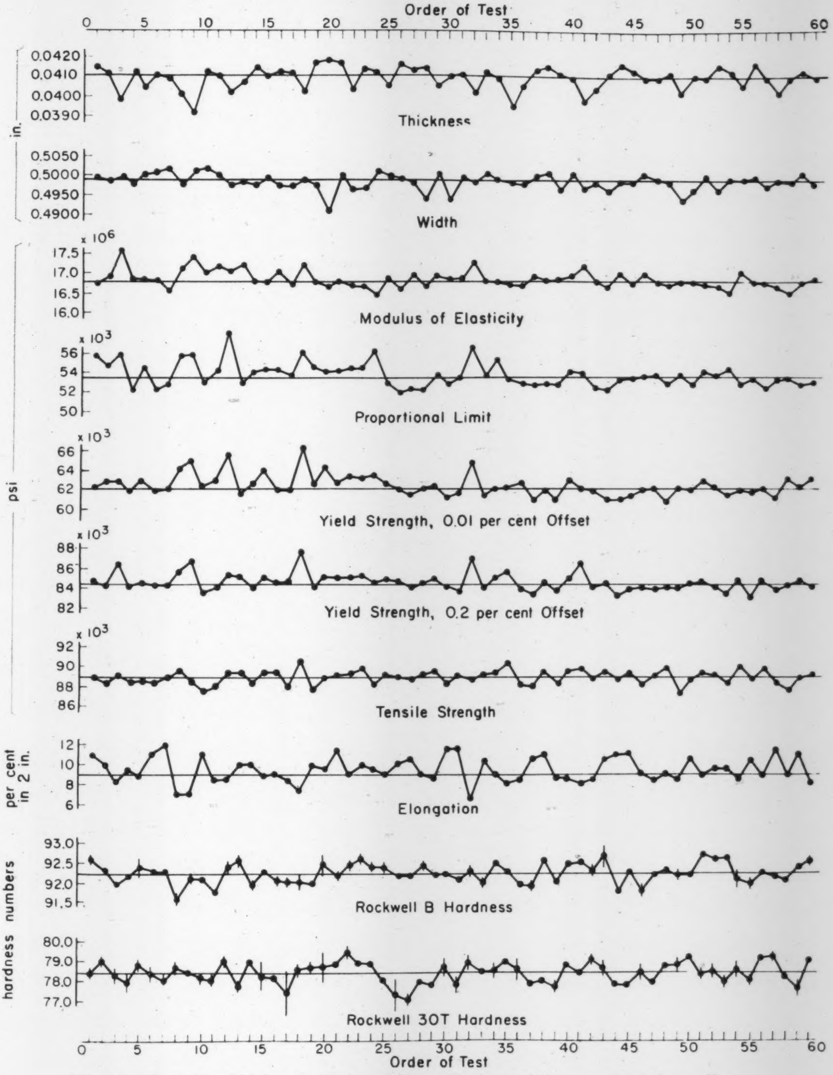

FIG. 2.—Individual Values in Order of Test. Horizontal lines are sample medians.

TABLE III.—SUMMARY OF MEAN SQUARE, OVER-ALL AVERAGE, $\hat{\mu}$, AND STANDARD DEVIATION, $\hat{\sigma}$, VALUES.

	Degrees of Freedom	Thickness	Width	Area	Modulus of Elasticity	Proportional Limit	Yield Strength		Tensile Strength	Elongation	Rockwell Hardness	
							0.01 per cent Offset	0.2 per cent Offset			B Scale	30T Scale
Columns and rows..	18	1.2828×10^{-6}	3.6444×10^{-6}	0.3102×10^{-6}	0.0961×10^{12}	2.075×10^{6}	2.392×10^{6}	2.649×10^{6}	0.892×10^{6}	3.9168	0.1411	0.3446
Columns, eliminating rows...	9	1.4611	6.4433	0.3434	0.1579×10^{12}	3.358	3.328	3.848	0.633	6.6460	0.0409	0.1357
Rows, eliminating columns....	9	0.6511	1.3478	0.1704	0.0167×10^{12}	0.612	1.855	0.998	1.262	0.7664	0.2475	0.4823
Milling lots........	5	0.0051	13.2056	0.0297	0.0202×10^{12}	2.018	0.767	0.235	0.205	0.7442	0.0237	0.2164
Residual..........	36	0.0105	2.3781	0.0055	0.0171×10^{12}	1.238	0.706	0.196	0.326	0.5118	0.0273	0.2534
$\hat{\sigma}$ (Residual).......		0.00010	0.00154	0.00007	0.13×10^{6}	1 113	840	443	571	0.72	0.17	0.50
$\hat{\mu}$ (Over-all average).		0.04082	0.49777	0.02032	16.793×10^{6}	53 466	62 234	84 326	88 774	9.46	92.22	78.37
$100\,\hat{\sigma}/\hat{\mu}$..........		0.24	0.31	0.34	0.77	2.08	1.35	0.53	0.64	7.61	0.18	0.64
$\hat{\sigma}$ (Total)..........		0.00063	0.00192	0.00032	0.20×10^{6}	1 249	1 107	944	699	1.25	0.25	0.53
$\hat{\mu} - 3\hat{\sigma}$ (Total).....		0.0389	0.4920	0.0194	16.19×10^{6}	49 719	58 913	81 494	86 677	5.71	91.47	76.78
$\hat{\mu} + 3\hat{\sigma}$ (Total).....		0.0427	0.5035	0.0213	17.39×10^{6}	57 213	65 555	87 158	90 871	13.21	92.97	79.96

TABLE IV.—SUMMARY OF RATIOS OF MEAN SQUARE TO RESIDUAL MEAN SQUARE VALUES.

	Ratio of Mean Square to Residual Mean Square										
	Thickness	Width	Area	Modulus of Elasticity	Proportional Limit	Yield Strength		Tensile Strength	Elongation	Rockwell Hardness	
						0.01 per cent Offset	0.2 per cent Offset			B Scale	30T Scale
Columns and rows	122.17**	1.53	56.82**	5.62*	1.68	3.39**	13.51**	2.74**	7.65**	5.17**	1.35
Columns, eliminating rows	139.15**	2.71*	62.91**	9.23**	2.71*	4.71**	19.63**	1.94	12.99**	1.50	0.54
Rows, eliminating columns	62.01**	0.57	31.21**	0.97	0.49	2.63*	5.09**	3.87**	1.50	9.07**	1.90
Milling lots	0.48	5.55**	5.45**	1.18	1.63	1.09	1.20	0.63	1.45	0.87	0.85
Between lots of 10	0.05	1.82	0.98	0.39	0.18	0.07	3.83	0.35	0.39	0.02	1.55
Between lots of 20	0.38	10.83**	10.45**	0.95	4.99*	4.05	1.22	0.46	2.75	0.08	0.00
Lots of 10 *versus* lots of 20	0.29	13.95**	12.69**	1.72	2.77	0.62	0.60	0.02	3.42	3.33	2.46
Within 20's (*B versus E*)	1.71	0.89	2.93	0.31	0.03	0.66	0.30	1.35	0.10	0.90	0.02
Within 20's (*A versus C*)	0.00	0.27	0.20	2.55	0.17	0.02	0.04	0.96	0.61	0.02	0.24

* Exceeds the tabulated value of the *F* ratio (**5**) corresponding to a significance level of 0.05.

** Exceeds the tabulated value of the *F* ratio (**5**) corresponding to a significance level of 0.01.

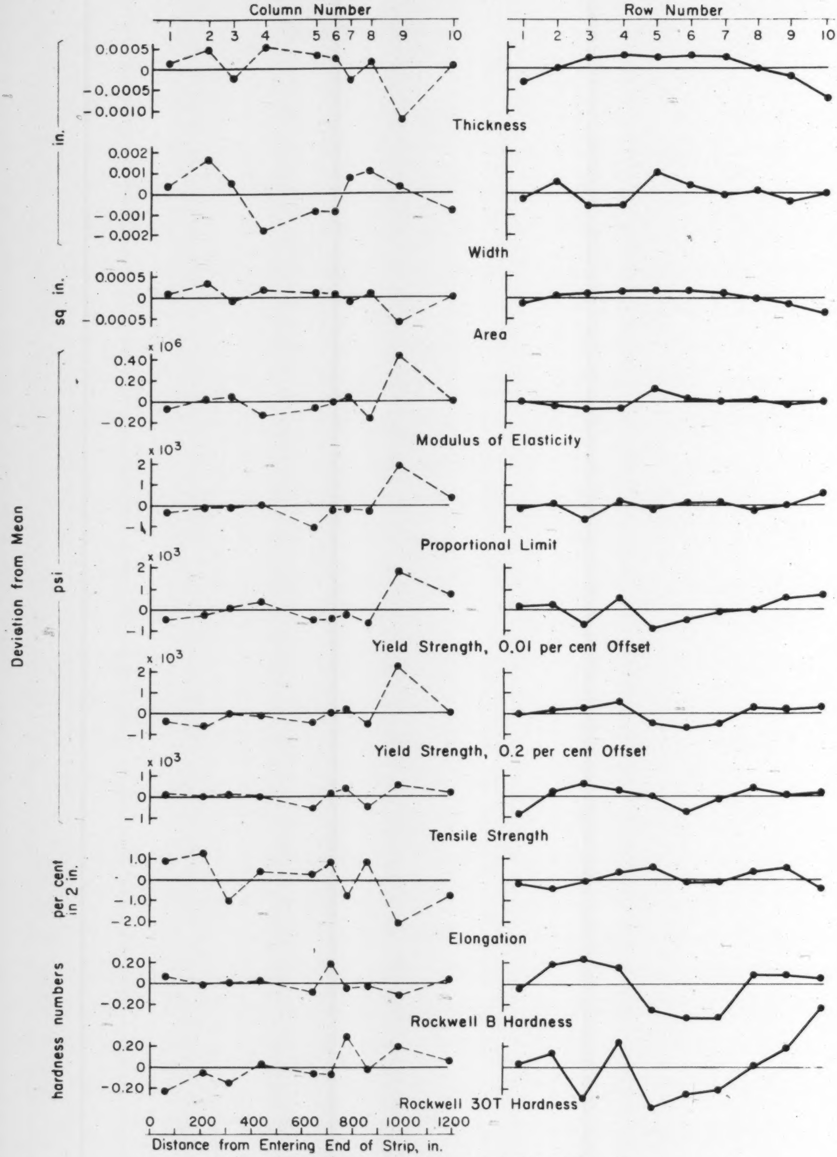

FIG. 3.—Estimates of Column and Row Effects.

portional limit, yield strength at 0.01 per cent offset, and yield strength at 0.2 per cent offset are high. In fact, the significant variation among columns for modulus of elasticity, proportional limit, yield strengths, and elongation appears to be due mainly to the thinness of the specimens in column 9. Tensile strength has a similar pattern of column effects, but the variation is not statistically significant (Table IV).

Estimates of the row effects, on the right side of Fig. 3, show the systematic variation of thickness across the strip. A comparison of the two charts for thickness shows that the variation across the strip is not as large as the variation along the length of the strip, though it is statistically significant (Table IV). The patterns of variation for the mechanical properties do not appear to be closely related to the pattern of variation for thickness, nor are they consistent within themselves. The amount of variation in thickness across the strip is apparently not large enough to mask the effects of other causes of variation, which may include differences in the final ready-to-finish grain size, the surface condition, and possibly in prior history.

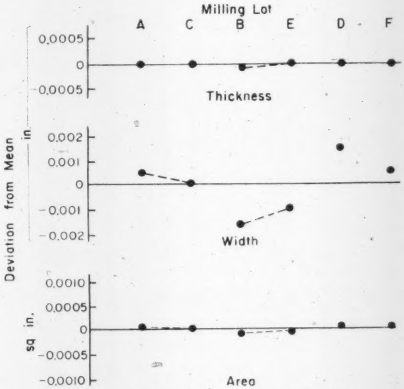

FIG. 4.—Estimates of Milling Lot Effects.

Tensile strength varies significantly with respect to rows, though the column variation was not significant (Table IV). The Rockwell hardness charts show more variation in row effects than in column effects; the variation for Rockwell hardness, B scale, is significant. The variation of yield strength at 0.2 per cent offset is more significant than that at 0.01 per cent offset (Table IV), though the actual variation of the row effects of the former is smaller. This result is due to the smaller residual mean square for yield strength at 0.2 per cent offset (Table III).

TABLE V.—VARIABILITY OF WIDTH IN MILLING LOTS.

	Variance	Average Variance
Lots of 10:		
D	0.64×10^{-6}	1.30×10^{-6}
F	1.97×10^{-6}	
Lots of 20:		
$(A + C)$	2.55×10^{-6}	3.44×10^{-6}
$(B + E)$	4.32×10^{-6}	

F Ratio, $\frac{3.44}{1.30} = 2.65$.

Variations in Milling Lots:

Table IV summarizes the comparisons made among and within milling lots. The characteristics that were affected significantly were width and area: among milling lots, between lots of 20, and between lots of 10 and lots of 20. Figure 4, which includes charts for thickness, width, and area only, shows that the specimens in the lot of 20 comprising the (pseudo) milling lots *B* and *E* were markedly narrower than the others. It is not clear whether this condition is related to the inclusion of 20 specimens instead of 10 in one milling lot or not, since the specimens in the (pseudo) milling lots *A* and *C* have widths that do not deviate markedly from those in lots *D* and *F*, which include only ten specimens each.

A statistical test (*F* test) indicates that the average variance of width for the milling lots of 20 is greater than the average variance for the milling lots of 10 (Table V); the large variance of lot ($B + E$) contributes importantly to the difference. A plot of width against stacking order shows more variability in the large milling lots but no important trends.

Thus there is some indication that small milling lots are preferable to large milling lots. However, the results of the analysis of variance (Table IV) give little evidence that the mechanical properties of the specimens are influenced significantly by the milling procedure.

Relative Precision of Measurements:

A comparison of the residual mean squares in Table III for proportional limit, yield strengths, and tensile strength is interesting. The mean squares for yield strength at 0.2 per cent offset and tensile strength are not significantly different. However, the mean square for yield strength at 0.01 per cent offset is significantly larger than those for yield strength at 0.2 per cent offset and tensile strength, while the mean square for proportional limit is larger yet. Since these properties were all determined from the same group of specimens, the differences may reflect dissimilarities in the precision of the measured load values for the four characteristics. Thus it may be that the load values for proportional limit were determined less precisely than those for yield strength at 0.01 per cent offset even though they were both read to ±5 lb. Likewise the Rockwell hardness 30 T numbers appear to be determined less precisely than the numbers on the B scale though the hardness numbers were all read to ±0.1.

Relationships Among Properties:

The variation in thickness shown in Fig. 3 is inherent in the method of manufacture of the phosphor bronze strip. Crown, that is, an increase in thickness from edge to center, is associated with bending of the rolls while variation along the length is associated with attempts to control the thickness within prescribed limits by increasing or decreasing the gap between the rolls on the basis of running thickness measurements made during cold reduction of the strip. The latter results in a series of transverse ridges and troughs which has a similar pattern for all ten blanks in any given column (Fig. 3).

Inspection of the column effects in Fig. 3 indicates that there is an association between the variation in thickness and the variation in the mechanical properties. The plots for modulus of elasticity, proportional limit, the two yield strengths, and tensile strength are almost direct images of each other. The column effects of these properties vary inversely with thickness, even though the direct effect of thickness is taken out by the mode of their computation. Except for the elastic modulus, this inverse relationship between thickness and the mechanical properties listed is in accordance with general metallurgical theory that increasing amounts of cold working, that is, reduction from a heavier to a lighter gage, should result in an increase in the value of the remaining four mechanical properties. Such theory also indicates that the elongation values should vary directly with thickness. The latter association was observed in the column effects but not in the row effects.

This absence of association in the row effects may be due to the fact that the systematic variation in thickness across the strip is not due to varying amounts of cold working but is present throughout the manufacture of the strip. While the crown imparted during processing prior to scalping is mostly removed by the latter operation, additional crown is, of course, rolled into the strip during subsequent cold working.

The plots of the row effects for the tensile and elastic properties show no direct association with thickness. Therefore, it appears to be varying amounts of cold working or per cent reduction in the material, indicated by the variations in thickness along the length, rather than the more or less uniform variation across the width that contribute significantly to the variation in many of the mechanical properties.

An interesting feature is the absence of any distinct relationship between the two hardness numbers, from either Fig. 2 or Fig. 3. Likewise, there is no strong association of either of the hardness numbers with the mechanical properties. This does not, of course, of itself invalidate the usual assumption of the existence of such relationships for gross changes from temper to temper.

The mechanical properties may be regarded as parameters which partially describe the load-strain curve for a specimen. The existence of some cross-relationships indicate some redundancy in the descriptive parameters. Further study of this matter might be useful.

CONCLUSIONS

Specimen thickness was found to vary systematically across the strip and to vary markedly from one position to another along the length. The specimens from one length (No. 14) were considerably thinner than those from other lengths and also showed corresponding extreme values for the column effects on the mechanical properties, excepting hardness numbers (Fig. 3).

Variability along the length of the strip was found to be statistically significant for most of the mechanical properties. Variability across the strip was found to be less significant except for tensile strength and the Rockwell hardness B scale numbers and does not appear to be associated with the variation in thickness.

It was found that the milling operation does not have a significant effect on the mechanical properties even though there is a significant difference among milling lots with respect to width. The variability of the width of the specimens in the larger milling lots was found to be significantly greater than that in the smaller milling lots.

The experiment also provides a comparison of the precision of load values used in computing proportional limit, the yield strengths, and tensile strength as well as a comparison of the precision of the two hardness numbers.

The results of this experiment indicate the amount of variability in the dimensions and mechanical properties that may be expected among specimens from a single strip of phosphor bronze. The variability of specimens from different strips would be even greater. Consideration should be given to the choice of the mechanical property used to compare lengths within a strip or different strips, since some properties are determined more precisely than others.

Reversed bending fatigue tests are now being made on specimens from this same strip of phosphor bronze. One phase of this investigation will be the relationship between the mechanical properties of particular lengths and the fatigue life of specimens from the same lengths.

Acknowledgment:

The authors wish to express their appreciation to W. G. Bader who made all of the mechanical tests and read the

load-strain charts; to Misses B. A. Leetch and J. Zagrodnick for making computations not done on the IBM 650 Computer; to Misses M. S. Harold and S. J. Reed for their assistance in the computations done on the IBM 650 Computer; and to Miss Elsa Kerner for preparing the illustrations.

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DISCUSSION

MR. H. N. CUMMINGS[1] (*by letter*).—Although the mechanical properties investigated by the authors did not include fatigue strength, their paper points up very clearly the reason for one of the sentences in "A Tentative Guide for Fatigue Testing and the Statistical Analysis of Fatigue Data," which has just been issued as separate publication *ASTM STP No. 91-A*. The sentence referred to is:

> "It is recommended that a statistician be consulted if a more complete exploration of this situation is considered necessary."

The large amount of information obtained from only 60 specimens obviously resulted from the statistical design and analysis described in the paper, even though the analysis is too complicated for inclusion in the Tentative Guide referred to above. And the analysis of variability in fatigue strength could easily have been included in the study, if fatigue test data had been available.

As a technical report the paper admirably meets all requirements, excepting possibly in what seems to the writer to be a confusing use of the terms "column" and "row." Would the authors state whether or not there is any tie-in between, say, column 0111 on Fig. 1 and column 1, designation 0111 in Table I?

MR. R. J. CHRISTINE[2] (*by letter*).—The experimental design using a modified Latin square to investigate the variability of mechanical properties is quite involved mathematically. However, the conclusions and graphs showing individual values in order of test can be readily appreciated.

This rather elaborate experiment confirms what technical mill-control personnel puts up with every day and it is most enlightening to realize the ramifications of random sampling in the mill. The next problem would of course be a solution as to why some of these physical properties seem to fluctuate out of proportion to others and how to control them.

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[1] Consulting Engineer, Curtiss-Wright Corp., Propeller Division, Caldwell, N. J.

[2] Technical Supervisor, Torrington Division, American Brass Co., Torrington, Conn.

It has been our experience that Rockwell hardness values for tempers over hard are relatively insensitive and often misleading when compared with tensile or yield strength. Surface effects are probably responsible for the lack of correlation between scales with varying loads.

The row effects can be attributed to the built-in crown throughout manufacture, as explained. The column effects are more subtle and rolling conditions during one or several passes would have an extremely large effect. Mill speed, lubrication, heating of rolls to change crown and screw-down changes, would all be factors. In addition, the uniformity of the interanneal throughout the coil would influence the deformation and rate of work hardening.

Internal soundness was not mentioned, but the elongation is usually a good indication of relative cross-section soundness.

One point that certainly should be emphasized as a result of this study is the folly of tightening up on mechanical property limits without a realization of the factors involved and the extent of variability.

Miss M. N. Torrey (*author's closure*).—In answer to Mr. Cummings, the variability in fatigue strength for this particular lot of phosphor bronze strip is now being studied and will be the subject of another paper which we hope to present before ASTM Committee E-9 on Fatigue. Mr. Cummings is correct in assuming that there is a tie-in between the columns shown in Fig. 1 and those shown in Table I. As pointed out in the text, the 100-ft length of phosphor bronze strip was divided into 17 lengths each of which was designated by the first two digits of a six digit number. Thus a specimen designated by the number 011101 was taken from the first length (01). Each length in turn was divided into 11 columns by cutting the strip normal to the rolling direction. The position of these columns in the strip is designated by the third and fourth digits (01 to 11). Finally each column selected for test was divided into 10 rows by shearing parallel to the length (rolling direction) and the position of each of these ten rows in their respective columns was designated by the last two digits (01 to 10). Thus the sample referred to in the second line of Table I by the first four digits of the designation, for example, 0111, would refer to any blank selected from the lower left hand column shown in Fig. 1. If the last two digits that identify the row, for example 01, were added, then the full designation would be 011101 and this would be the top blank shown in the left hand column of Fig. 1. In Table I, the first four digits are therefore shown in line 2 across the top of the table to identify the ten columns selected at random, while the two digits in the left hand column complete the identification of the individual specimen blanks. The numbers 1 to 10 in line 1 of this table have no significance other than to indicate the order in which the randomly selected columns were grouped to form the Latin square.

We appreciate the comments of Mr. Christine and hope that others will be made aware of the significance and usefulness of studies such as we have reported in setting up specification requirements.

MICROSTRUCTURAL OBSERVATIONS PERTINENT TO THE TEMPERING AND TEMPER BRITTLENESS OF STEEL*

By W. P. Clancy[1] and M. R. Norton[1]

Synopsis

Utilizing the maximum resolution of the visible light microscope, the authors have demonstrated the growth of ferrite grains in an SAE 3140 steel tempered for varying times and at various temperatures below the lower critical temperature. The evidence presented supports the view that "upper nose isothermal temper embrittlement" is nothing more than a manifestation of the change in impact properties accompanying the normal changes in microstructure which occur during the fourth stage of tempering. Reported ferrite grain diameters of the order of 2μ are consistent with those previously published for plain carbon steels.

Etching practices associated with temper brittleness studies are discussed. It is pointed out that significant structural details may be overlooked because of these practices.

Woodfine (**1**,**2**)[2] has suggested that the increase in Charpy impact transition temperature with increase in time at temperatures immediately below the lower critical temperature is caused by an increase in ferrite grain size. He offered this as a probable explanation of the so-called "upper nose temper embrittlement" observed in alloy steels. Jaffe and Buffum (**3**) indicated, however, that no increase in grain size was observed in the SAE 3140 steel used in their investigation. Powers, in discussing Jaffe and Buffum's results (**3**),[3] noted their lack of evidence of ferrite grain growth, which seemed contrary to the data of others and to Gilman's (**10**) theory of crack propagation in crystal aggregates.

Hyam and Nutting (**4**) have demonstrated, with microstructural evidence, that, in fact, ferrite grain size does increase with increasing tempering temperature and increasing time at temperature in plain carbon steels having carbon contents varying from 0.1 to 0.9 per cent. They also suggested that the process of simultaneous growth of ferrite grains and carbide particles be called the fourth stage of tempering. Owen and co-workers (**5**) and Frazier, Boulger, and Lorig (**6**), investigating ship steels, Hodge, Manning, and Reichhold (**7**), investigating mild steels, with and without nickel, Burns and Judge (**8**), investigating a 0.24 per cent plain carbon steel, obtained data which show

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* Presented at the Sixty-first Annual Meeting of the Society, June 22–27, 1958.

[1] Materials Research Laboratory, Ordnance Materials Research Office, Watertown, Mass.

[2] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 930.

[3] *Transactions*, Am. Inst. Mining and Metallurgical Engrs., Vol. 209, p. 1316; *Journal of Metals*, Oct., 1957.

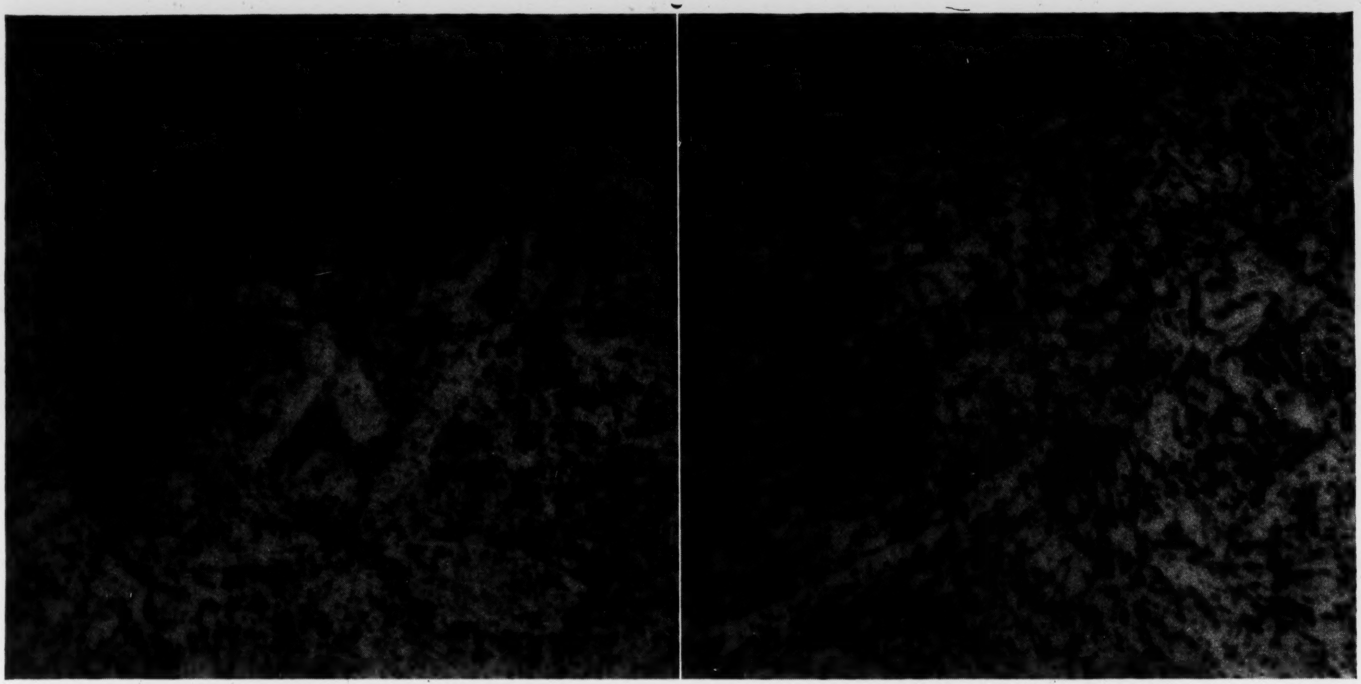
TABLE I.—CHEMICAL COMPOSITION.

Steel Designation	Composition, per cent								
	Carbon	Manganese	Silicon	Sulfur	Phosphorus	Nickel	Chromium	Molybdenum	Vanadium
SAE 3140 steel	0.39	0.79	0.30	0.028	0.015	1.26	0.77	0.02	...
SAE 1020 steel	0.21	0.44	0.11	0.024	0.007	trace	0.02	trace	...
4.5 Nickel steel	0.34	trace	0.34	0.027	0.005	4.52	trace	trace	...
Manganese-vanadium steel	0.14	1.30	0.20	0.021	0.02	nil	0.025	trace	0.115
Manganese-nickel-chromium-molybdenum steel	0.28	1.57	0.32	0.027	0.017	0.63	1.06	0.49	...
Nickel-chromium-molybdenum steel	0.27	0.76	0.32	0.017	0.010	3.29	1.54	0.49	...
Nickel-chromium steel	0.35	0.87	0.22	0.022	0.029	1.16	0.50	trace	...

TABLE II.—HEAT TREATMENT AND IMPACT PROPERTIES.

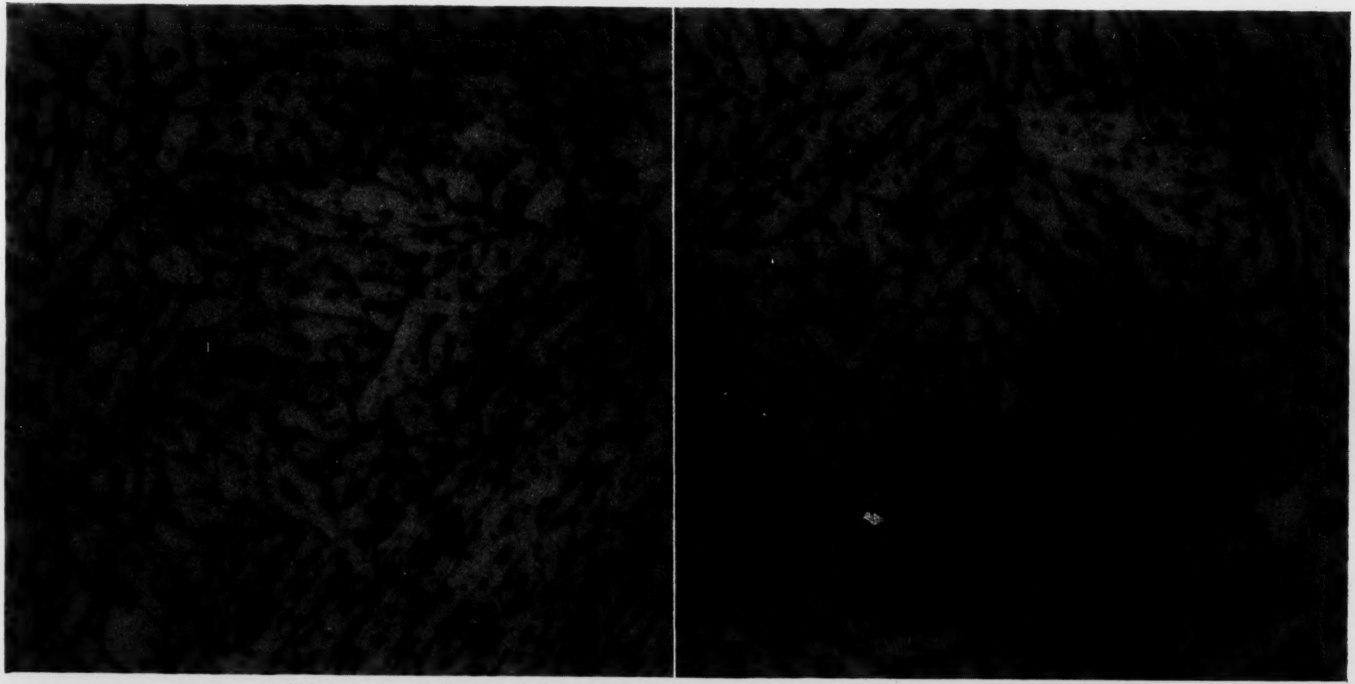
Steel Designation	Austenitizing Treatment	Tempering and Embrittlement Treatments	Impact Energy, ft-lb	Testing Temperature, deg Cent	Impact Transition Temperature, deg Cent[a]
SAE 3140 steel	900 C, 1 hr, water quenched	700 C, 5 min, water quenched	...	...	−78
		700 C, 8 hr, water quenched	...	...	−40
		675 C, 5 min, water quenched	...	...	−81
		675 C, 240 hr, water quenched	...	...	−14
		650 C, 5 min, water quenched	...	...	−72
		650 C, 48 hr, water quenched	...	...	−35
		675 C, 1 hr, water quenched	...	...	−78
		675 C, 1 hr, water quenched, plus 500 C, 240 hr, water quenched	...	...	+25
SAE 1020 steel	1000 C, 1 hr, transferred to bath at 725 C, held for 30 min, water quenched	680 C, 1 hr, water quenched	7.5	+20	...
		680 C, 1 hr, furnace cooled at rate 17 C per hr	58.0	+20	...
4.5 Nickel steel	840 C, ½ hr, water quenched	650 C, ½ hr, water quenched	35.8	−40	...
		650 C, ½ hr, furnace cooled at rate 17 C per hr	25.8	−40	...
Manganese-vanadium steel	900 C, 1 hr, water quenched	650 C, 1 hr, water quenched	127.0	−40	...
		650 C, 1 hr, furnace cooled	8.9	−40	...
Manganese-nickel-chromium-molybdenum steel	900 C, 3 hr, water quenched	650 C, 4½ hr, water quenched	74.9	−40	...
		650 C, 4½ hr, furnace cooled	42.4	−40	...
Nickel-chromium-molybdenum steel	940 C, 4 hr, oil quenched	650 C, 4 hr, water quenched	63.7	−40	...
		650 C, 4 hr, water quenched, plus 455 C, 500 hr, water quenched	7.2	−40	...
Nickel-chromium steel	870 C, 1 hr, water quenched	600 C, 1 hr, water quenched	98	0	...
		600 C, 1 hr, water quenched, plus 455 C, 500 hr, water quenched	12	0	...

[a] Transition temperatures listed are Jaffe and Buffum's uncorrected values (**3**).



(*a*) Tempered at 650 C, 5 min, water quenched. (*b*) Tempered at 700 C, 5 min, water quenched.

FIG. 1.—SAE 3140 Steel. Etchant: Aqueous picric acid, 3 min. Objective: APO 108, N.A. 1.6 (× 3500).



(*a*) Tempered at 650 C, 48 hr, water quenched. (*b*) Tempered at 700 C, 8 hr, water quenched.

FIG. 2.—SAE 3140 Steel. Etchant: Aqueous picric acid, 3½ min. Objective: APO 108, N.A. 1.6 (× 3500).

that an increase of transition temperature occurs with increase in equiaxed ferrite grain size. Baeyertz, Craig, and Bumps (**9**), investigating a steel of eutectoid composition, containing nickel, presented evidence of an increase in transition temperature with increase in ferrite grain size which also involved a change in grain morphology.

Because experimental verification of ferrite grain growth during the tempering of an SAE 3140 steel should provide basic information relative to (*a*) the tempering process and (*b*) upper nose embrittlement, the present study was undertaken. Incidental to the use of metallographic techniques, additional data were acquired concerning etching effects, for the material under investigation as well as for some other steels.

MATERIALS AND PROCEDURES

Tempered structure studies were made using the SAE 3140 steel for which Charpy impact transition data are available from the work of Jaffe and Buffum (**3**). Etching studies were conducted on specimens of other steels as well as the one just cited. Chemical compositions for all steels are included in Table I. Heat treatments and pertinent Charpy impact data are given in Table II. All steels, with the exception of the SAE 1020, were quenched to 100 per cent martensite. The SAE 1020 steel was treated to produce patches of martensite in a ferrite matrix.

All specimens were mechanically polished. In order to investigate tempered structures, specimens were etched for 3 to 4 min in a 0.5 per cent aqueous solution of picric acid. To check etching effects, specimens were immersed, for varying times, in either of two reagents, namely, ethereal picric acid – Zephiran chloride (**13**) and 0.5 per cent aqueous solution of picric acid.

Microstructures were observed and photographed with a visible light microscope, employing Apochromat 60, 0.95 N.A. and Apochromat 108, 1.6 N.A. objectives. To secure maximum resolution, an interference filter, peaked for 4360 Å, and spectroscopic plates, type II-O, were used in conjunction with the 1.6 N.A. objective.

Ferrite grain sizes in the SAE 3140 steel were estimated by an intercept method. Three representative fields of each specimen were photographed at 3500 diameters. A total of six measurements was made for each field along three sets of two mutually perpendicular lines. The average ferrite grain diameter, d, reported herein, is the value obtained from the relation

$$d = \frac{D}{M} = \frac{l}{Mn}$$

where D is defined as the mean distance between ferrite boundaries intercepted along a straight line by both ferrite-ferrite boundaries and ferrite-carbide boundaries at the magnification M, l is the total intercept length, and n is the number of ferrite grains and carbides intercepted. The value of n was obtained by counting the total number of boundaries intercepted.[4]

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[4] To be precise the value of d as reported is an average of the ferrite grain size and carbide particle size. In order to obtain a value for d which would represent the average grain diameter alone, one would have to count the number of ferrite grains only and subtract the length intercepted by each carbide particle from the total intercept length. The length intercepted by each carbide particle is not always easily determined because some of the carbides are so small that their measurement becomes a problem of splitting hairs. The authors did not feel that it was necessary to resort to such a laborious procedure to obtain values which attempt to show nothing more than relative differences in grain size. It is a much simpler task to count the number of boundaries intercepted, arbitrarily counting those carbides having no open centers and located in ferrite grain boundaries as ferrite-ferrite boundaries.

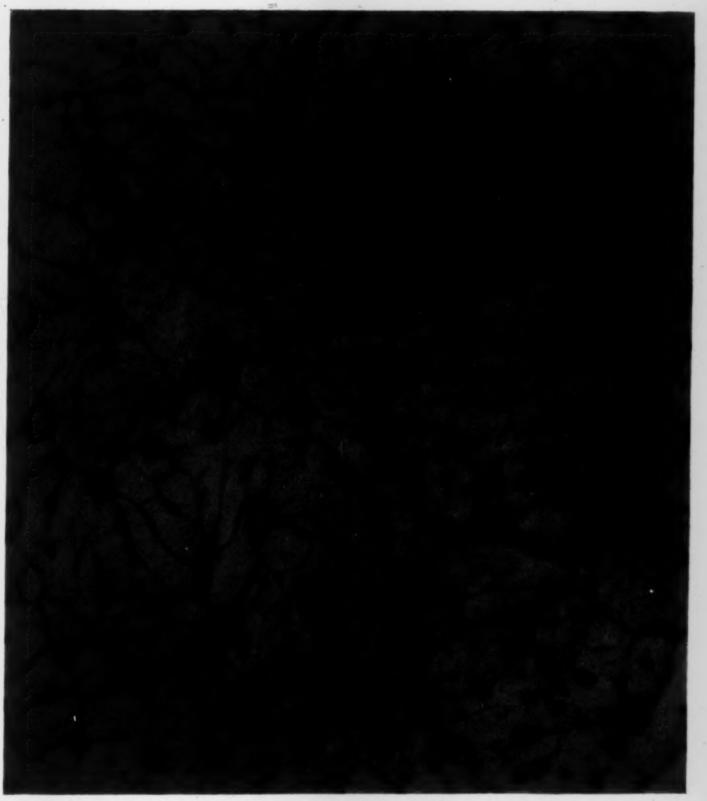

FIG. 3.—SAE 3140 Steel. Etchant: Aqueous picric acid, 3 min. Tempered at 675 C, 240 hr, water quenched. Objective: APO 108, N.A. 1.6 (× 3500).

RESULTS AND DISCUSSION

Tempered Structures:

The structures of SAE 3140 specimens tempered at temperatures within the range of 650 to 700 C are shown in Figs. 1 to 3 inclusive. Comparison of Fig. 1(*a*) (650 C for 5 min) with Fig. 1(*b*) (700 C for 5 min) shows a subtle change in structure. In Fig. 1(*a*) are areas which closely resemble, in shape, the original martensite needles. This general appearance is absent in Fig. 1(*b*). Carbide particles in both cases, however, are manifest as extremely small, black pinpoints. The specimen tempered at 675 C for 5 min displayed a structure intermediate between that of Fig. 1(*a*) and Fig. 1(*b*), and is not illustrated.

The structure produced by a 48-hr temper at 650 C is quite comparable with that resulting from an 8-hr temper at 700 C, as may be seen in Figs. 2(*a*) and (*b*). Appreciable growth of carbide particles is evident in each case. Unlike the structures resulting from short tempering times, those illustrated in Figs. 2(*a*) and (*b*) display pronounced ferrite grains. Although some areas contain elongated ferrite grains, other areas contained ferrite grains which are beginning to assume an equiaxed shape. The suggested tendency for ferrite grains to change from the elongated, primitive shape to that of equiaxed outlines with increasing tempering time, is confirmed in Fig. 3. The latter illustrates the structure obtained after holding for 240 hr at 675 C. Again, with an increase in grain size, there is an accompanying increase in carbide size. It should be noted that carbides are, in general, associated with ferrite grain boundaries at each stage of grain growth, and that for equivalent ferrite grain sizes obtained at two different temperatures (Figs. 2(*a*) and (*b*)), the carbide particle size appears to be the same.

TABLE III.—GRAIN SIZE MEASUREMENTS.

Tempering Temperature and Time	650 C, 48 hr	675 C, 240 hr	700 C, 8 hr
Average ferrite grain diameter, *d*, mm	0.0019	0.0023	0.0018
Uncorrected transition temperature, deg Cent[a]	−35	−14	−40

[a] From reference (**3**).

Measurements of ferrite grain sizes together with associated Charpy impact transition temperatures are presented in Table III. The data indicate clearly that an increase in transition temperature accompanies an increase in grain size and that, within experimental error,[5] material having the same grain size, obtained under different tempering conditions, will exhibit the same transition temperature. No value representing change of transition temperature with respect to change in grain size, using the data in Table III, is presented. A meaningful calculated value would require a determination of the average ferrite grain diameter exclusive of the carbide particle size.[4]

The important result to be considered is that ferrite grain growth does take place at temperatures below the lower critical temperature in SAE 3140 steel, contrary to the report of Jaffe and Buffum (**3**) and consistent with Woodfine's (**1,2**) reasoning. The present experimental data lend further support to the view that the upper nose embrittlement is nothing more than a manifestation of the change in impact properties accompanying the normal changes in microstructure which occur during the fourth stage of tempering.

Of secondary importance is the fact that growth in grains having a diameter of the order of magnitude of 2 μ can be observed without employing electron microscopy, as was thought necessary by Fisher and Boyce (**9**)[6] and by Libsch, Powers, and Bhat (**20**). The accuracy of the authors' grain size measurements is believed to be at least as good as those made by investigators heretofore employing plastic replicas for this purpose. Indeed, Hyam and Nutting (**4**), as a result of their own ferrite grain size studies, note the limitation of the plastic replica technique to reproduce distinct grain boundaries. The present authors recognize, however, that a higher degree of accuracy, potentially, can be obtained with electron microscopy than with visible light microscopy, providing an adequate replica technique is employed.

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[5] The accuracy of the transition temperature data was estimated to be ±5 C.

[6] *Transactions*, Am. Inst. Mining and Metallurgical Engrs., Vol. 191, p. 1064; *Journal of Metals*, Nov. 1951.

Etching Effects:

The evidence described above would not have been observed had the authors adopted a practice very common to workers studying the microstructural aspects of temper brittleness, namely, the light repolishing of the etched metal surface. Indeed, many investigators may have destroyed significant structural details in this manner.

Etching to detect temper brittleness caused by tempering at temperatures below 600 C has become almost a standard procedure. Picric acid base reagents, with and without special additive agents, have been used successfully, although there has been some divergence of opinion concerning the response to etching by steels of varying chemical composition (**1,13,14,16,17**). It is generally reported that when two specimens of a given material, one in the unembrittled condition and one in the embrittled condition, are etched simultaneously for a given time and then repolished for about 1 min, the embrittled specimen displays outlines which are either absent in the unembrittled specimen, or only faintly visible. Furthermore, the outlines usually observed are equivalent to the boundaries of the former austenite grains. As a consequence, experiment and theory have been concerned with reactions which may occur at or near the prior austenite grain boundaries. Evidence that fracture tends to follow such boundaries has provided further incentive for seeking microstructural explanations of temper embrittlement in these areas. Ferrite grain boundaries, which are nearly completely wiped out during the repolishing, have received little consideration. Because the literature on temper brittleness continues to grow (**12**), the present authors believe it timely to suggest that greater emphasis on studies of ferrite grains and grain boundaries may be fruitful. In support of this thought, it is now proposed to review, briefly, some etching results observed over a period of years.

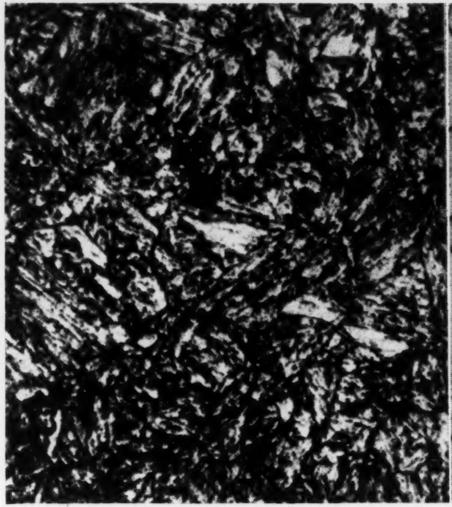
Figures 4 and 5 typify an etching effect which has been noted for each steel listed in Table I; that is, if one prolongs the etching time for the unembrittled specimen, it is possible to obtain an etched structure which resembles that of the embrittled counterpart (**15,19,20**). This is true both before and after a slight repolish.

Figure 4 illustrates the structure of the nickel-chromium steel, in Tables I and II, obtained under the conditions described in the captions. It should be noted that the structure in Fig. 4(*c*), the unembrittled specimen etched for 10 min, is essentially the same as that of Fig. 4(*e*), the embrittled specimen etched for 2 min.

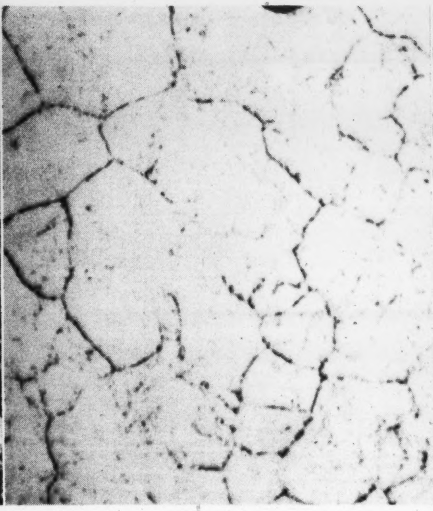
Figure 5 illustrates the structure of the manganese-nickel-chromium-molybdenum steel, in Tables I and II, obtained under the conditions described in the captions. It should be noted that the grain boundary outlines of the unembrittled specimen, Fig. 5(*g*), when etched for 30 min are as strongly delineated as are those of the embrittled specimen, Fig. 5(*a*), when etched for 10 min. In fact, after a slight repolish in each case, the grain boundary outlines in the unembrittled specimen appear to be heavier. Compare Fig. 5(*b*) with Fig. 5(*h*).

Figure 6 illustrates the structure of the 4.5 nickel steel, in Tables I and II, and typifies another etching effect, namely, the pronounced outlining of ferrite as well as prior austenite grains in the embrittled specimen (see Fig. 6(*b*)).

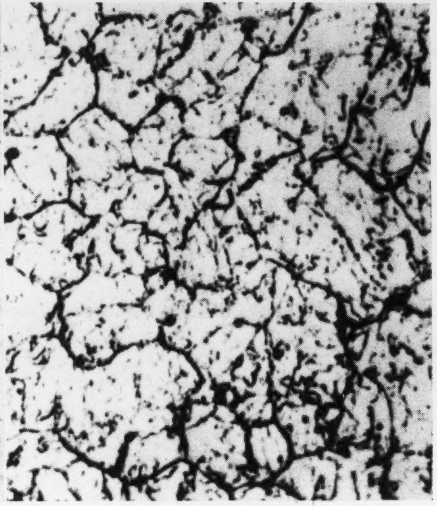
This case of heavy attack of ferrite grain boundaries in the 4.5 nickel steel is not the only instance of its kind. An SAE 1020 steel (see Tables I and II), treated so as to give a microstructure



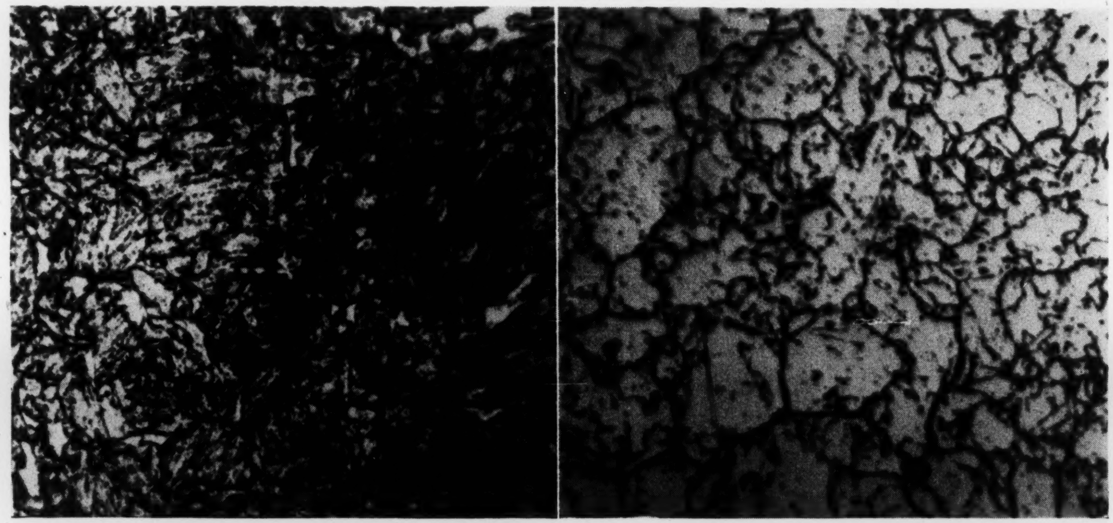
(*a*) Tempered at 600 C, 1 hr, water quenched, etched with ethereal picric acid – Zephiran chloride, 2 min.



(*b*) Same surface as (*a*) after a slight repolish.



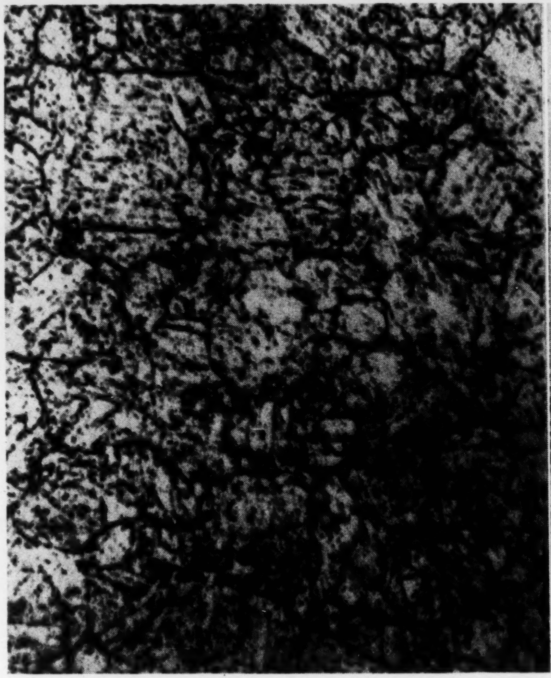
(*c*) Same specimen as (*a*) and (*b*), new surface, etched with ethereal picric acid – Zephiran chloride 10 min, plus a slight repolish.



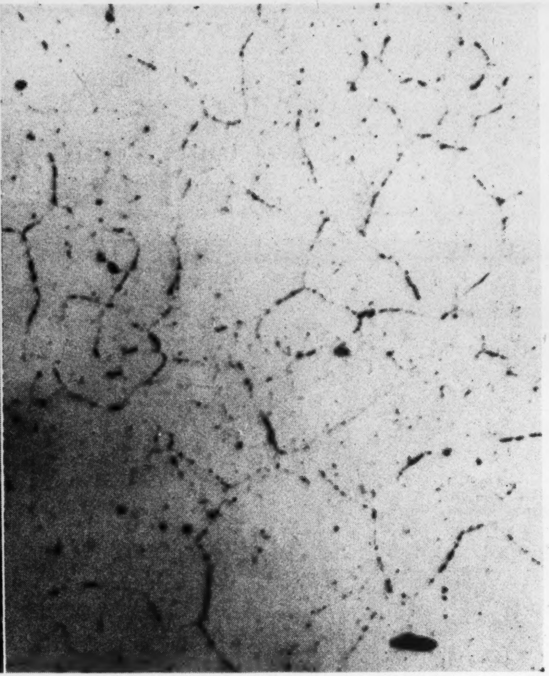
(*d*) Tempered at 600 C, 1 hr, water quenched, embrittled at 455 C for 500 hr, water quenched, etched with ethereal picric acid - Zephiran chloride, 2 min.

(*e*) Same surface as (*d*) after slight repolish.

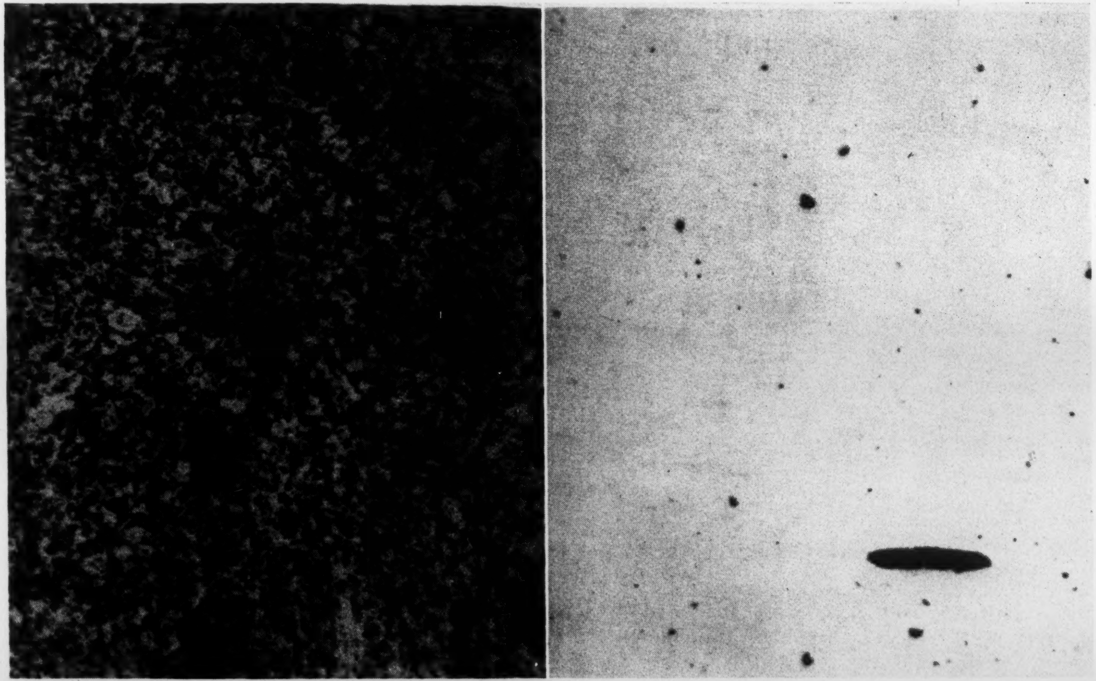
FIG. 4.—Nickel-Chromium Steel. Objective: APO 60, N.A. 0.95 (× 1000).



(*a*) Tempered at 650 C for 4½ hr, furnace cooled, etched with ethereal picric acid – Zephiran chloride, 10 min.



(*b*) Same surface as (*a*) after a slight repolish.

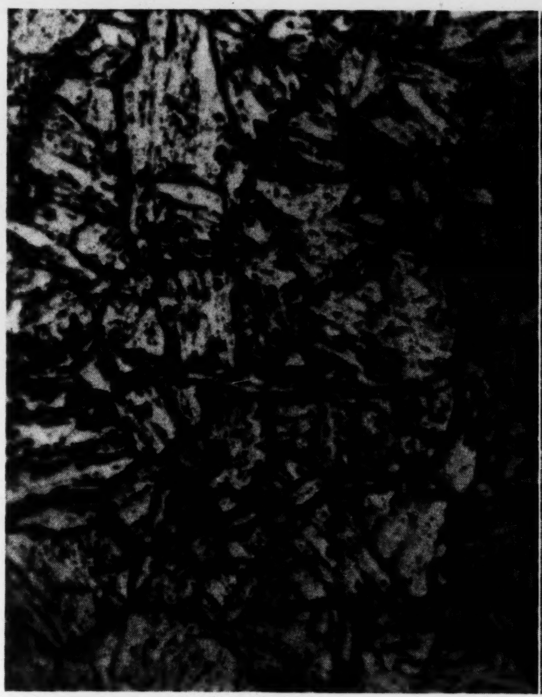


(*c*) Tempered at 650 C for 4½ hr, water quenched, etched with ethereal picric acid - Zephiran chloride, 10 min.

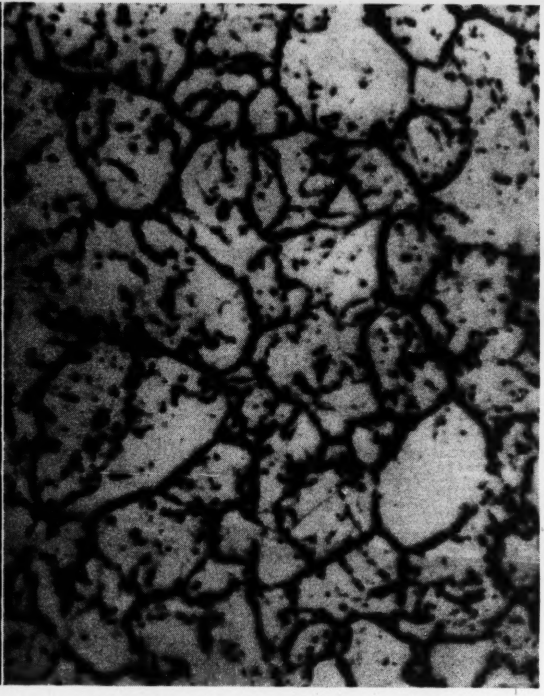
(*d*) Same surface as (*c*) after a slight repolish.

Fig. 5.—Manganese-Nickel-Chromium-Molybdenum Steel. Objective: APO 60, N.A. 0.95 (× 1000).

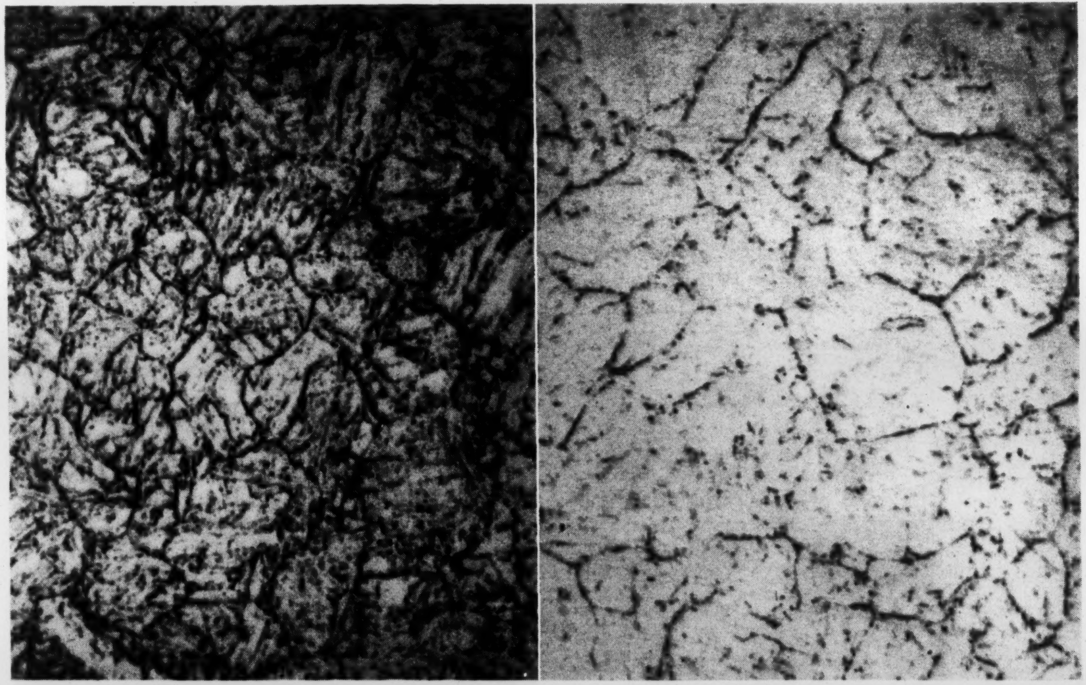
[Continued on pages 924 and 925.]



(*e*) Same specimen as (*a*), new surface, etched with ethereal picric acid – Zephiran chloride, 30 min.



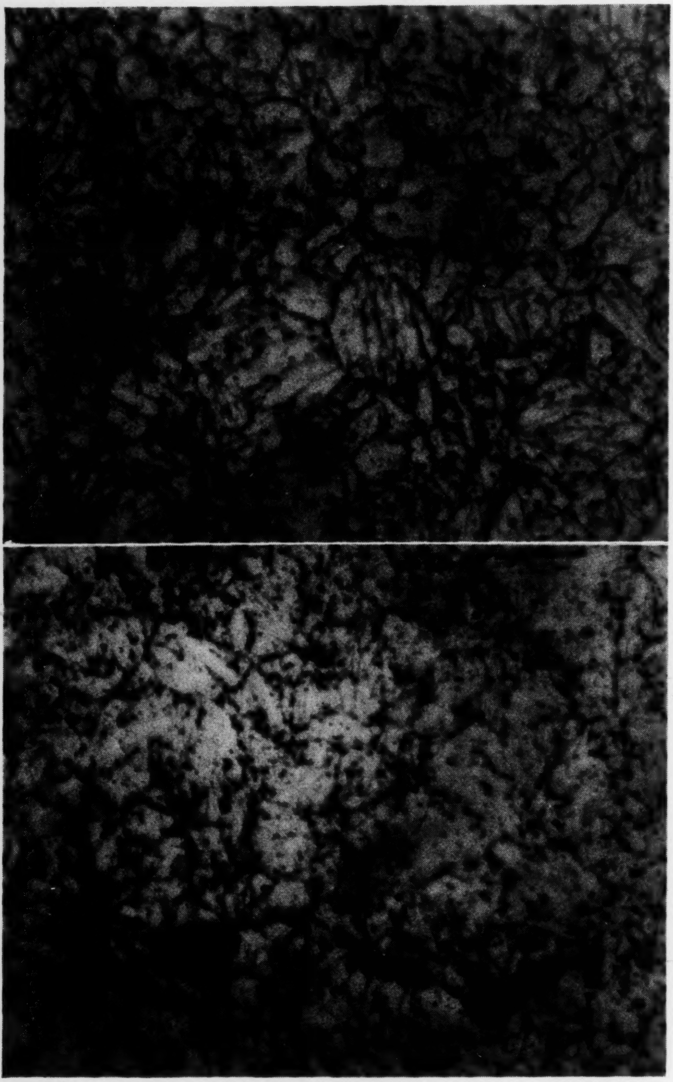
(*f*) Same surface as (*e*) after a slight repolish.



(*g*) Same specimen as (*c*), new surface, etched with ethereal picric acid - Zephiran chloride, 30 min.

(*h*) Same surface as (*g*) after a slight repolish.

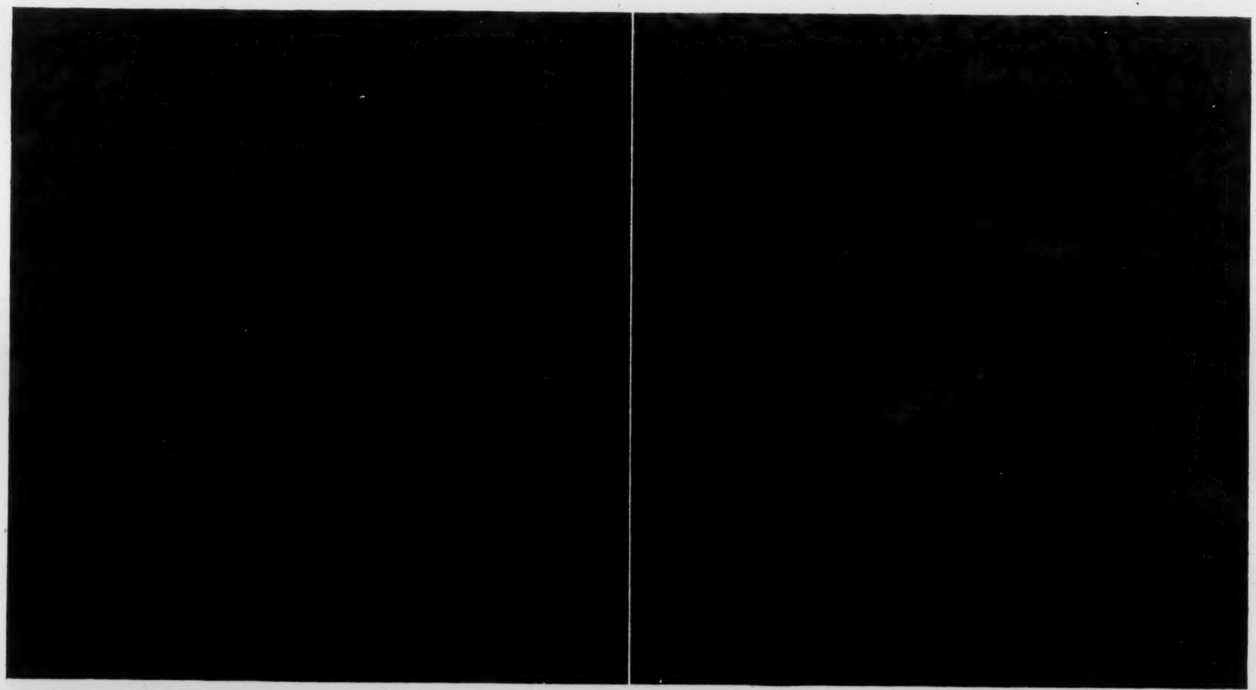
Fig. 5.—*Concluded.*



(*a*) 650 C, ½ hr, water quenched, etched with aqueous picric acid, 4 min.

(*b*) 650 C, ½ hr, furnace cooled at 17 C per hr, etched with aqueous picric acid, 4 min.

FIG. 6.—4.5 Nickel Steel. Objective: APO 60, N.A. 0.95 (× 1000).

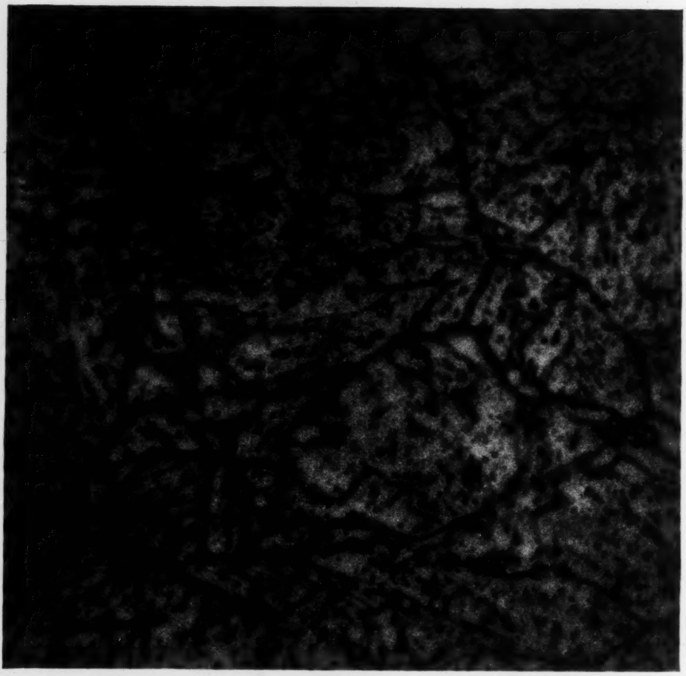


(*a*) Tempered at 675 C for 1 hr, water quenched, etched with aqueous picric acid, 3½ min.

(*b*) Same specimen as (*a*), new surface, etched with aqueous picric acid, 15 min.

FIG. 7.—SAE 3140 Steel. Objective: APO 108, N.A. 1.6 (× 3500).

[Continued on page 928.]



(*c*) Tempered at 675 C for 1 hr, water quenched, embrittled at 500 C for 240 hr, water quenched, etched with aqueous picric acid, 3½ min.

FIG. 7.—*Continued.*

consisting of islands of tempered martensite[7] among near-equiaxed ferrite grains (ASTM No. 7),[8] showed a higher rate of attack in aqueous picric acid at the primary ferrite grain boundaries for the furnace-cooled material than for the material water quenched from the temper.

It should be noted from Table II that the furnace-cooled material (Rockwell hardness B 59) has much better impact properties than the water-quenched material (Rockwell hardness B 84). At the 10 ft-lb level, the transition temperatures were −48 C and +27 C for the furnace-cooled and water quenched material, respectively.

The reason for the switch in the expected relative positions of the temperatures is beyond the scope of this paper. It is sufficient to note the fact that here is a plain carbon steel which, when furnace cooled, shows the expected higher rate of grain boundary attack commonly associated with furnace-cooled material and yet it has better impact properties than the water-quenched material. These etching results are contrary to those reported by Libsch, Powers, and Bhat (20) for a picric acid base etchant.

Figure 7 illustrates a number of additional etching effects for the same SAE 3140 steel which was discussed earlier relative to ferrite grain growth and which was shown structure-wise in Figs. 1 through 3. Figure 7(*a*), unembrittled, and etched with aqueous picric acid for $3\frac{1}{2}$ min, displays some heavy boundaries, possibly prior austenite boundaries. When etching time is increased to 15 min, however, the structure seen in Fig. 7(*b*) is revealed. Here, ferrite boundaries are as heavily outlined as the prior austenite ones. Figure 7(*c*) embrittled by holding for 240 hr at 500 C, and etched for $3\frac{1}{2}$ min, also depicts heavy outlines of both prior austenite and ferrite grains. Like Fig. 6(*b*), Fig. 7(*c*) illustrates clearly that the outlines of the prior austenite grain boundaries now actually comprise boundaries of many neighboring ferrite grains. Finally, Fig. 7(*c*), unlike Fig. 7(*b*), manifests strings of discrete carbide particles in both ferrite and prior austenite grain boundaries. The presence of these carbides, of the order of 0.3 μ in diameter, was confirmed by an electron microscope study of direct carbon extraction replicas.

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[7] The percentage of martensite areas was approximately 30.

[8] Methods for Estimating the Average Ferrite Grain Size of Low-Carbon Steels (E 89 - 52), 1958 Book of ASTM Standards, Part 3.

With reference to Figs. 1–3, 6, and 7, it should be recorded that, of the picric acid base etchants used, only aqueous picric acid was found satisfactory for studies of etched structures at higher magnifications and resolutions. The criticalness of etching may explain, in part, why published electron micrographs of temper embrittled steels (**1,17,18**) have not yet resolved the question: Is there a relation between microstructure and temper brittleness?

SUMMARY

The following points have been demonstrated:

1. Ferrite grain growth occurs in an SAE 3140 steel at tempering temperatures immediately below the lower critical temperature. This evidence indicates that "upper nose isothermal temper embrittlement" should be thought of as the change in impact properties which takes place concomitant with microstructural changes during the fourth stage of tempering.
2. The maximum resolution of the visible light microscope can be employed to detect microstructural features which are ordinarily considered beyond the capabilities of this instrument.
3. For temper brittleness studies, significant structural details are destroyed through the practice of lightly polishing after etching.
4. With sufficient etching time in etchants having a picric acid base, the same degree of etching attack found in embrittled specimens can be induced in unembrittled specimens of a given steel.
5. Upon etching with reagents having a picric acid base, ferrite grain boundaries, as well as prior austenite grain boundaries of both embrittled and unembrittled specimens, are heavily delineated. This is not surprising since, after tempering, prior austenite grain boundaries and ferrite grain boundaries are one and the same.

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DISCUSSION

Mr. Samuel Epstein.[1]—Do the authors claim that grain growth is the only reason embrittlement occurs in temper-embrittled steel?

Mr. W. P. Clancy (*author*).—A distinction must be made between the so-called upper nose embrittlement and the lower nose embrittlement. We suggest that upper nose embrittlement is due entirely to ferrite grain growth and consequently is irreversible whereas the lower nose embrittlement is due to some as yet unknown process which has been shown to be a reversible.

Mr. Epstein.—If the upper nose embrittlement is due to grain growth, why should it take longer etching to bring out the grain boundary in the unembrittled state than in the embrittled state?

Mr. Clancy.—Etching tests relative to the degree of etching attack *versus* etching time were made only on those specimens in a series concerned with the lower nose embrittling process. No etching time tests were made on those specimens which had been tempered at temperatures immediately below the lower critical temperature and which showed marked ferrite grain growth.

Mr. Epstein.—It would be of interest then to determine whether or not upper nose embrittlement, ascribed to grain growth, can be detected by longer etching in the same way as lower nose embrittlement.

Mr. Clancy.—The authors made no claim that lower nose embrittlement can be detected by longer etching time.

Mr. John M. Hodge.[2]—I only wish to comment that this work has needed to be done for a long time, and that it has certainly cleared up a very controversial aspect of the temper brittleness question.

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[1] Technical Advisor, Bethlehem Steel Co., Bethlehem, Pa.

[2] Research Consultant, Alloys, Applied Research Laboratory, United States Steel Corp., Monroeville, Pa.

THE EFFECTS OF STRAIN RATE AND TEMPER ROLLING ON THE STRAIN-AGING CHARACTERISTICS OF RIMMED, DEEP-DRAWING STEEL*

By D. H. Fisher,[1] R. L. Carlson,[2] and W. T. Lankford[3]

Synopsis

Experimental studies were performed to investigate the effects of strain rate and temper-rolling extension on the strain-aging characteristics of rimmed deep-drawing steel. The effects after aging times of 29, 150, and 800 hr were investigated for two amounts of temper-rolling extension (1.2 per cent and 2.3 per cent) at strain rates varying from 0.001 per sec to 0.4 per sec. Commercially temper-rolled material was used, and all tests were performed at room temperature.

The results of the study are summarized by plots that depict the variation of the stress-strain behavior with varying strain rate, amount of temper rolling, and aging time. The significance of the indices used to describe the results is discussed in terms of the history of the specimens and the strain rate. Yield-point phenomena, work-hardening characteristics, and the uniform elongation are found to be affected by history and strain rate, and emphasis is directed toward relating the observed behavior to the mechanics of plastic flow.

When annealed low-carbon sheet steel is deep drawn, strain lines, known as "stretcher strains," sometimes appear on the surface. These stretcher strains result from nonhomogeneous plastic deformation as the steel is stretched. In a tension test specimen, this localized deformation is correlated with the appearance of a double yield point, the yield point elongation, and the formation of Lüders bands.

Temper rolling is a common commercial method of eliminating or reducing the severity of yield point phenomena in sheet material (**1**).[4] The amount of temper-rolling extension impressed is small enough ($\frac{1}{2}$ to 2 per cent) to produce no marked decrease in ductility, and yet sufficient to be effective. In the freshly temper-rolled condition, the steel forms smoothly. During storage of some steels, however, there will be a gradual return of yield point phenomena and also an increase in the strength and a decrease in ductility. The changes in mechanical properties that occur with time have been described as strain aging. Cottrell (**2**) and his co-workers have made outstanding contributions to our understanding of the changes in yield point phenomena resulting from strain aging. The increase in strength and de-

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* Presented at the Sixty-first Annual Meeting of the Society, June 22–27, 1958.

[1] Principal Mechanical Engineer, Battelle Memorial Institute, Columbus, Ohio.

[2] Assistant Division Chief, Battelle Memorial Institute, Columbus, Ohio.

[3] Chief Research Engineer, Specialty Products, U. S. Steel Corp., Monroeville, Pa.

[4] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 942.

crease in ductility that occur with strain aging have recently been associated by Hundy with a mechanism which he called "strain-age hardening" (3).

The amount of temper rolling necessary to control stretcher strains in a low-carbon steel depends on the amount of yield point elongation present in the annealed condition. Tension tests are frequently conducted in the laboratory to determine the amount of yield-point elongation and also the aging characteristics of the material. In many cases, however, the rates of straining are not representative of the rates encountered in the press room. Although a yield point elongation may not be observed at slow strain rates, it may be quite extensive at strain rates equivalent to those of the press room. This sensitivity of yield point phenomena to strain rate has been illustrated by several experimental studies (**4**, **5**, **6**).

The experimental studies summarized by this paper were designed to investigate the effects of strain rate and temper rolling on the strain-aging characteristics of a rimmed deep-drawing steel. Special emphasis is directed toward describing the changes that occur in the yield point phenomena, the rate of work hardening, and the uniform elongation during aging.

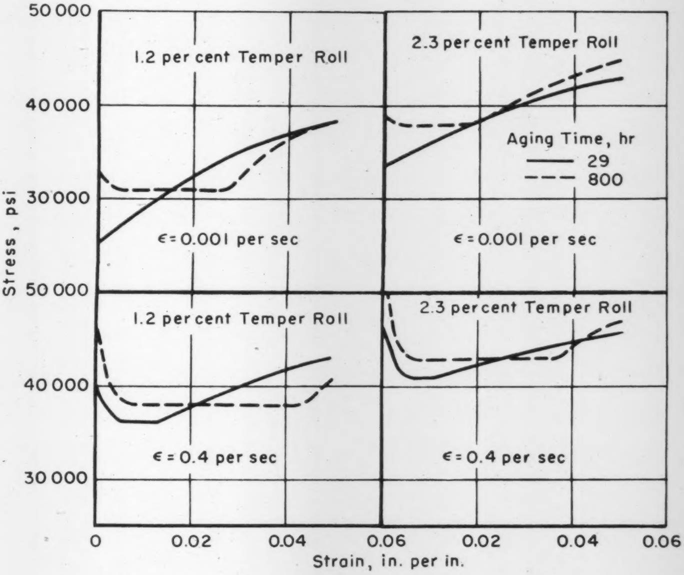

Fig. 1.—Yield-Point Phenomena for Various Test Conditions.

Experimental Procedure and Material

The equipment used to perform the tests conducted in this investigation has been described previously (**7**). The equipment consists of a hydraulic press used in conjunction with a subpress designed to convert the downward movement of the ram to a tensile force on a sheet specimen. The head rate of the press was adjusted to obtain strain rates from 0.001 to 0.4 per sec on a sheet tensile specimen with a uniform 4-in. gage section.

A Sanborn recorder was used to record the load and the strain during the tension tests. The load and strain were measured by SR-4 strain gages applied to a dynamometer ring and extensometer loops, respectively.

The strain rate values were calculated from the permanent records of strain *versus* time. The recorded load and strain measurements were used in conjunction with the following equations to obtain true stress - true strain data:

$$\sigma = \frac{L}{A_0}(1 + \epsilon) \qquad \ldots\ldots\ldots\ldots (1)$$

$$\delta = \ln (1 + \epsilon) \qquad \ldots\ldots\ldots\ldots (2)$$

where:

σ = true stress, psi
δ = true strain, in. per in.
L = applied load, lb
A_0 = original cross-sectional area, sq in., and
ϵ = nominal strain, in. per in.

The strain rates, as calculated from the recorder chart, represent the rate of deformation throughout the plastic region of the curve (after yielding). The strain rate in the elastic region (prior to yielding) was estimated to be on the order of five times slower than in the plastic region.

The composition in per cent of the rimmed steel used in this investigation was: carbon = 0.08, manganese = 0.39, phosphorus = 0.014, sulfur = 0.034, silicon = 0.006. The grain size was ASTM Nos. 7 to 8.[5]

All of the test material was obtained from 20-gage sheet (nominally 0.038 in. thick) sheared from a single coil in which the temper-rolling extension was varied by adjusting the mill setting during rolling of the coil. An automatically indicating temper-mill extensometer was used as a guide for making the mill adjustments, but the actual extensions were determined from the change in distance between scribe marks originally 25 in. apart. The temper-rolling was carried out at Gary Sheet and Tin Mill of U. S. Steel Corp. on a four-high temper mill having 22 in. diameter work rolls. Temper-rolling extensions of 1.2, 1.9, and 2.3 per cent were obtained. Only the results for the 1.2 and 2.3 per cent extensions are presented in this paper, since they clearly indicate the trends observed.

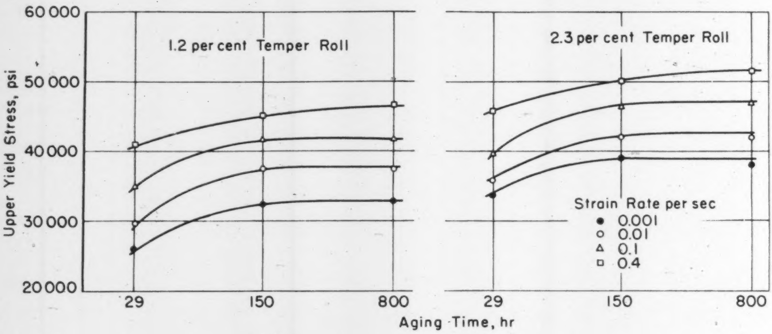

FIG. 2.—Upper Yield Stress *versus* Aging Time.

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[5] Methods for Estimating the Average Ferrite Grain Size of Low-Carbon Steels (E 89 – 52), 1955 Book of ASTM Standards, Part 1, p. 1690.

After aging for periods of 29, 150, and 800 hr, tension tests were conducted at strain rates of 0.001, 0.01, 0.1, and 0.4 per sec. All experiments were performed at room temperature. The minimum aging time (29 hr) represents processing, storage, and machining time accumulated before testing. During shipment the sheet material was stored at a temperature of about −100 F.

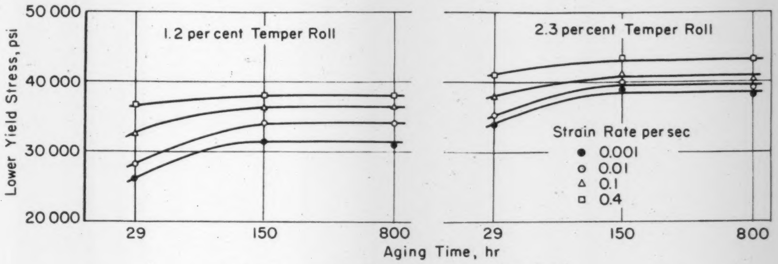

FIG. 3.—Lower Yield Stress *versus* Aging Time.

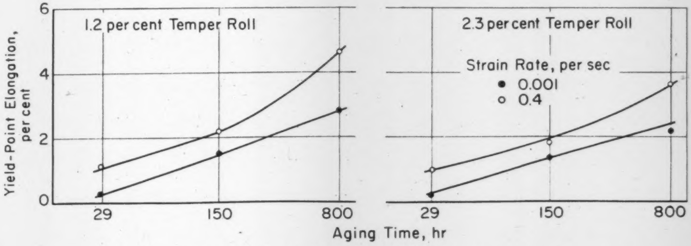

FIG. 4.—Yield-Point Elongation *versus* Aging Time.

DISCUSSION OF RESULTS

The results obtained in this investigation are summarized in Figs. 1 to 7. The curves presented indicate the variation of the mechanical properties with strain rate, amount of temper rolling, and aging time. Each of the plotted points of Figs. 2 to 7 represents data from four tests. The variation of individual values from the plotted values will be noted in the discussion that follows. A discussion of the effect of test conditions will be presented in four sections: initial yielding; work hardening; uniform elongation; ultimate strength.

Initial Yielding:

The variations in the yielding behavior for several of the testing conditions are shown in Fig. 1. Representative curves for extreme aging periods (29 and 800 hr) and the extreme strain rates (0.001 per sec and 0.4 per sec) are shown. The curves for intermediate conditions lie between the extremes. From the curves, the effects of strain rate, amount of temper rolling, and strain-aging time on the "front" or small strain part of the stress-strain curve can be noted.

The curves of Fig. 1 indicate that aging and increasing strain rate raise the front of the stress-strain curve, and increase the magnitude of the yield point elongation. It would appear that straining at a rapid strain rate has an effect similar to that produced by aging.

The effect of temper rolling is also illustrated in Fig. 1. Increasing the degree of temper rolling from 1.2 per cent to 2.3 per cent has raised the front of the stress-strain curve. This is the effect that would be expected from increased cold working. It can also be noted that the extent of the yield point elongation has been reduced.

The effect of aging time and strain rate on the upper and lower yield point stresses is shown in Figs. 2 and 3. The variation of individual values from the plotted points is ±1500 psi or less. Both the upper and lower yield point stresses increase with increasing strain rate, and the general level of the curves for 2.3 per cent temper rolling is higher than that for 1.2 per cent temper rolling. Most of the increase observed with aging appears to have occurred during the first 150 hr. Using these values as indices of aging would tend to suggest that the aging mechanism had essentially become "stabilized" after about 150 hr.

Another possible index of aging is the yield point elongation. The variation of individual values of yield-point elongation from the plotted points of Fig. 4 is ±0.25 per cent of strain or less. Since the variations observed between strain rates (for example, 0.001 to 0.1 per sec) were of this order, only the data for extreme strain rates are plotted. Values for intermediate rates lie between the curves shown.

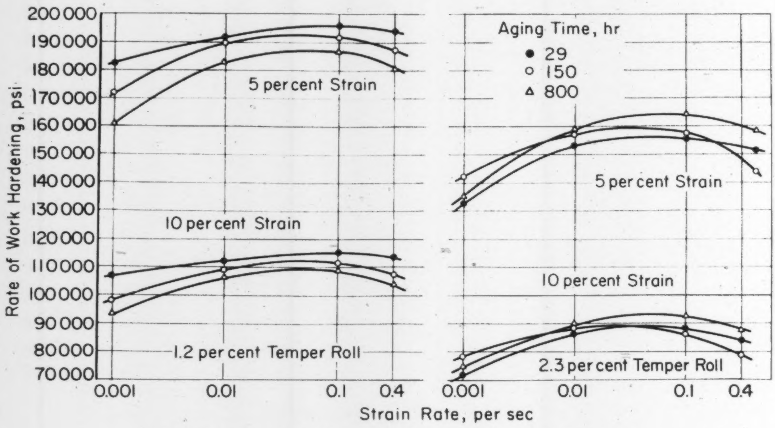

FIG. 5.—Rate of Work Hardening *versus* Strain Rate.

The curves of Fig. 4 suggest that the effect of aging on yield elongation continued throughout the period investigated. Since the amount of yield elongation is more directly related to the deep-drawing problem of inhomogeneous straining than are the yield stresses, the pattern of aging indicated in Fig. 4 must be considered as more revealing than that indicated by Figs. 2 and 3. Where there appears to be no advantage gained in increasing the degree of temper rolling to 2.3 per cent in Figs. 2 and 3, the results of Fig. 4 indicate a distinct advantage. For the 2.3 per cent material, both the rate and the amount of increase due to aging are less than for the 1.2 per cent material.

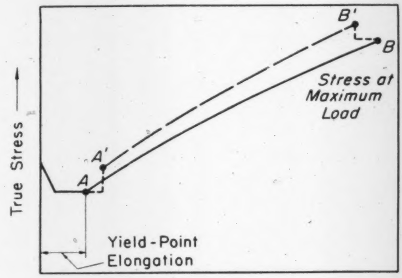

FIG. 6.—Effect of Strain Rate.

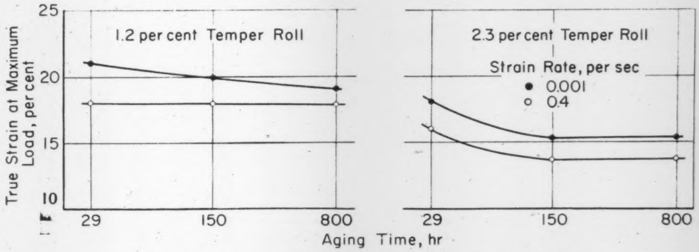

FIG. 7.—True Strain at Maximum Load *versus* Aging Time.

On the basis of the data obtained on uniform elongation (see Fig. 7), the advantage of increasing the amount of temper rolling from 1.2 to 2.3 per cent to improve the yielding behavior is questionable. The advantage of a smaller yield point elongation after aging is offset by the reduced amount of retained ductility. In this respect, it is interesting to note that the maximum amount of temper rolling in commercial practice is usually less than 2 per cent.

The curves of Fig. 4 indicate that the magnitude and the rate of change in yield point elongation with aging is dependent on strain rate. If only slow strain rates (of the order of 0.001 per sec) were used, it would appear that the yield point elongation had been "eliminated" for aging times less than about 29 hr. The curves for a strain rate of 0.4 per sec, however, suggest that a yield elongation would probably be observed even at zero time for rapid strain rates.

Work Hardening:

In the preceding sections, emphasis was directed toward describing the effect of strain rate, aging time, and temper-rolling extension on the properties associated with initial yielding. The response of a material beyond this stage is also important, however. The onset of instability or necking is, in fact, related to the rate at which a material work hardens. It can be shown (**8**) that instability occurs when the rate of work hardening equals the stress. In other words the stress at maximum load is equal to the rate of work hardening, or

$$\frac{d\sigma}{d\delta}\bigg/_{\delta_{\max}} = \sigma_{\delta_{\max}} \ldots\ldots\ldots\ldots (3)$$

where $\delta_{\max}$ is the true strain at maximum load.

A computation of the rate of work hardening associated with certain amounts of strain and various aging times and degrees of temper rolling was facilitated in this study by the fact that the true stress - true strain data could be represented by the empirical relation:

$$\sigma = B\delta^n \ldots\ldots\ldots\ldots\ (4)$$

The rate of work hardening at various strains could then be readily computed from the relation

$$\frac{d\sigma}{d\delta} = n\frac{\sigma}{\delta} \ldots\ldots\ldots\ldots\ (5)$$

The rate of work hardening was computed by the use of Eq 5 for several of the test conditions, and the results are presented in Fig. 5. The plotted points represent values computed for four tests. The variations of the individual values from the plotted points were ±3000 psi or less. From the plots the effects on the rate of work hardening of strain, temper-rolling extension, aging time, and strain rate are shown.

The rate of strain hardening decreases with increasing strain. This is a normal behavior; it is of interest to note, moreover, that the forms and relative positions of the curves are essentially unchanged by increasing strain. The apparent reason for the observed form of the curves will be discussed later.

Increasing the degree of temper rolling from 1.2 to 2.3 per cent resulted, for the given amounts of strain, in decreases in the rates of work hardening. This suggests that though the work hardening produced by temper rolling is not uniform through the thickness of the specimen, the effect on the rate of work hardening is similar to what would be expected for uniform prestraining.

The effect of increasing aging time observed for the material temper rolled 1.2 per cent was to decrease the rate of work hardening. This trend may be associated with the fact that although the stress at maximum load increased only slightly with increasing aging time, the rise in the front of the stress-strain curve (the lower yield point) due to strain aging was quite significant. This could produce a general decrease in the rate of work hardening as the curve from the lower yield point to the maximum load rotates clockwise with increasing aging time.

The trend noted above was not observed for all test conditions. For specimens temper rolled 2.3 per cent, there is a tendency for a reversal of the ordering with respect to aging time. This reversal appears to be inconsistent with the other results. It may, however, be associated with the strain-age hardening mechanism suggested by Hundy (3) to explain decreased ductilities and increases in strength after long aging times in temper-rolled low-carbon steel. The suggested mechanism postulates that a general hardening arises from precipitation effects, possibly associated with the precipitation of very small nitride particles. Such particles would impede dislocation movement after the initial yielding process and thereby result in an increase in tensile strength and a decrease in ductility.

In terms of temper-rolling extension, Hundy concluded that effects of strain aging for small amounts of temper rolling are manifested primarily as changes in yield stress and yield point elongation. The effects depend primarily on the anchoring of dislocations by interstitial solute atoms as suggested by Cottrell. For larger degrees of temper rolling or for longer aging times, on the other hand, tensile strength and ductility are markedly affected—the strength increasing and the ductility decreasing. The latter effects are attributed to the strain-age hardening mechanism. If this explanation is correct, the changes in stress-strain behavior that occur with strain aging, then, are the result of two phenomena: dislocation anchoring by interstitial atoms, which affects the front of the stress-strain curve, and strain-age hardening which affects the back of the stress-strain curve.

Returning now to the curves of Fig. 5, the tendency for an increase in the rate of work hardening with aging time may be explainable in terms of strain-age hardening. This hypothesis appears to be supported by Hundy's observation that the strain-age hardening effect increases with increasing temper-rolling extension and increasing aging time. The reversal in the change in the rate of work hardening in increasing the temper-rolling extension from 1.2 to 2.3 per cent may represent a transition in which the influence of dislocation anchoring by solute atoms is replaced in importance by strain-age hardening.

The general and consistent effect of strain rate is to first increase and then decrease the rate of work hardening with increasing strain rate. These variations appear to be related, in turn, to the observed effect of strain rate on the yield elongation, the lower yield point, and the stress and strain at maximum load. This follows from the fact that the slope of the stress-strain curve in the region of interest is strongly influenced by these four values. This is illustrated in Fig. 6. The curve *AB* represents the back part of the stress-strain curve for the slowest strain rate (0.001 per sec). As the strain rate increases, the changes indicated in Fig. 6 and in the tabulation below are observed:

	Factor	Change With Increasing Strain Rate	Effect on Rate of Work Hardening
No. 1...	Lower Yield Stress........	Increase	Decrease
No. 2...	Yield Elongation..........	Increase	Increase
No. 3...	Stress at Maximum Load..	Increase	Increase
No. 4...	Strain at Maximum Load..	Decrease	Increase

This tabulation suggests that, of the four factors cited, only the change in the lower yield stress would tend to contribute a decrease in the rate of work hardening with increasing strain rate. If this reasoning is correct in essence, the curves of Fig. 5 indicate that factors 2, 3, and 4 are predominant for rates between 0.001 and 0.01 per sec. At about 0.01 per sec, factor 1 is becoming important. Beyond about 0.1 per sec, it would appear the factor 1 has become, relatively, most important.

The tendency for the rate of work hardening to decrease at the faster strain rates is of interest in terms of instability or necking. Prior to necking, the inequality $\frac{d\sigma}{d\delta} > \sigma$ holds. As the strain rate increases beyond 0.1 per sec, the stress at a strain of say, 5 per cent, would increase and the rate of work hardening would decrease. This indicates that the inequality tends to become an equality, or that the instability condition can be expected to be satisfied by a sufficient increase in strain rate. This effect should manifest itself as a decrease in the strain at maximum load (uniform elongation) with increasing strain rate. This was observed in the tests conducted. These results will be discussed in the next section.

Uniform Elongation:

A summary of the data on uniform elongation or the true strain at maximum load is presented in Fig. 7. Each of the plotted points represents data from four tests. The variation of the actual test data from the plotted points was of the order of ±1 per cent of strain. Since this was of the order of the change observed in going from one strain rate to the next (0.001 to 0.01 per sec, for example), only the values for the fastest and the slowest rates have been plotted to indicate the trends. The data for the intermediate rates conformed to the pattern shown and ranged in values between the extremes indicated by the plots.

Figure 7 indicates that δ_{max} decreased with increasing strain rate. In the previous section on work hardening, it was suggested that this decrease should follow from the fact that as the strain rate increases, particularly beyond 0.1 per sec, the stress at a given strain increases and the rate of work hardening decreases.

The curves of Fig. 5 suggest that the rate of work hardening would continue to decrease significantly at strain rates greater than 0.4 per sec. For the 2.3 per cent material at a strain of 10 per cent, an extrapolation of the rate of work hardening curves to strain rates of about 10 per sec suggests that values of about 60,000 to 70,000 psi might be anticipated. Since the stresses, which are of the order of 55,000 psi at 10 per cent strain for a strain rate of 0.4 per sec, would be expected to be significantly larger for a strain rate of 10 per sec, it can be seen that δ_{max} would probably be less or only slightly larger than 10 per cent. This follows from the requirement that $\frac{d\sigma}{d\delta} > \sigma$ for stable, uniform elongation.

The correlation between the strain at maximum load and the value of n of Eq 4 was fair. For the material temper rolled 1.2 per cent, the values of δ_{max} ranged from about 1 to 2 per cent of strain lower than the n values. For the 2.3 per cent material the values of δ_{max} were both higher and lower than the n values. The general difference was observed to be of the order of 1 per cent of strain.

The equality of n and δ_{max} depends on both the accuracy of the selection of the point at which slope of the load curve becomes zero, and the validity in this region of Eq 4. Since both of these determinations are subject to error, it does not appear to be possible to determine with finality which of the values, δ_{max} or n, represents the uniform elongation more accurately.

Increasing the degree of temper rolling extension from 1.2 to 2.3 per cent resulted in a significant decrease in the strain at maximum load. Although a part of this decrease could be attributed to the decrease in available ductility accompanying increased amounts of cold work, the decrease observed is in excess of what would be expected. This suggests that the loss in ductility accompanying aging has been increased by the additional amount of temper rolling extension. This is in accord with Hundy's observations which were discussed above.

Ultimate Strength:

The ultimate strength or the stress at maximum load was found to be sensitive to the rate of straining. An increase of about 8 per cent was observed for increases in strain rate of from 0.001 to 0.4 per sec. This amount of increase occurred for all aging times and both degrees of temper rolling. Over the same range of strain rates, Manjoine (5) observed an increase of about 6 per cent for a low-carbon steel. It should be noted, however, that for rates above about 1 per sec, increases in the ultimate strength with increases in strain rate were observed by Manjoine to be much more marked.

No significant or consistent difference in the ultimate strength was observed for the two amounts of temper-rolling extension or for increased aging times. Work by Hundy (3) indicates that for temper-rolling extensions of less than about 2 per cent, the increase in the ultimate strength is hardly significant after an aging time of about 2000 hr. (The maximum aging time in this study was 800 hr.) For a temper-rolling extension of 2 per cent, the increase was observed by Hundy to be about 500 psi or about 1 per cent. The data of Hundy, then, support the observed insensitivity of the ultimate strength to temper-rolling extensions and aging times of the range investigated in the present study. For longer aging times, and particularly for greater degrees of temper-rolling extension, however, Hundy's studies indicate that significant increases could be expected.

SUMMARY

The findings discussed in this paper can be summarized as follows:

1. The changes that occur with strain aging in the yield point stress and the yield point elongation are dependent on the strain rate and the degree of temper-rolling extension.
2. The data for the slowest strain rate could be used as evidence that yield point phenomena were "eliminated" during a short period immediately after temper rolling. Data for the faster rates, however, suggest that yield point phenomena could have been observed immediately after temper rolling for sufficiently rapid strain rates.
3. Increasing the degree of temper-rolling extension from 1.2 per cent to 2.3 per cent resulted in a slower return of yield-point phenomena with aging. This advantage was offset, however, by a significant decrease in ductility.
4. The change in the rate of work hardening appears to be influenced by two strain-aging phenomena. The first, associated with anchoring of dislocations by interstitial solute atoms, appears to raise the front of the stress-strain curve and result in a decrease in the rate of work hardening. The second, referred to as strain age hardening, may be responsible for a second, opposing effect, which causes an increase in the rate of work hardening with aging. The first effect appears to be dominant for the smaller degree of temper rolling. The second effect seems to be becoming dominant for the greater degree of temper roll.
5. The rate of work hardening passes through a maximum and finally decreases with increasing strain rate. The latter tendency coupled with the trend for the stress-strain curve to rise with increasing strain rate can be expected to result in decreased total elongation. This follows from the fact that for uniform stable flow, the true stress must be less than the rate of work hardening. The data on uniform elongation tend to support this conclusion.
6. The uniform elongation decreased with increasing aging time and with the increased degree of temper-rolling extension.
7. The ultimate strength increased slightly with increasing strain rate. For the range of aging times and temper-rolling extensions investigated, however, the ultimate strength showed no significant change for a given strain rate.

Acknowledgment:

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TOUGHENING HIGH-STRENGTH STEEL BY WARM WORKING*

By E. J. Ripling[1]

Synopsis

The poor toughness associated with SAE 1340 steel at high strength levels can be greatly improved (two- to sevenfold) by warm working. This is accompanied by an appreciable increase in yield strength, a slight increase in tensile strength and ductility, as well as by a mild loss in hardness. A somewhat smaller toughness improvement, accompanied by a smaller hardness loss, is found in warm-worked SAE 4340 steel.

A series of recent publications (**1–7**)[2] has shown that high-strength steels as well as many other toughness-deficient metals (annealed steels (**1**), pure zinc (**3**), titanium alloys (**7**)) are suffering from a strain correctible type of brittleness (rheotropic brittleness). Brittleness in these metals was found to be partially eliminated by cold working the material; and the brittleness was most completely eliminated if the cold working or prestraining operation (usually stretching) was followed by an overaging treatment. Since some ductility is sacrificed in performing the prestraining operation, the corrective treatment itself exhausts some ductility. This loss was found to be minimized if the mean stress during prestraining had the largest possible negative value. This suggests that elevated temperature deformation (simultaneous straining and aging) by a compressive type of deformation, such as extrusion, should produce the toughest possible product. Further, elevated temperature working has the added advantage of softening the metal so that the deformation is more easily performed.

The study described herein was undertaken in order to determine the properties (especially tensile ductility and impact toughness) of warm-worked high-strength steels. The largest portion of the work was conducted on SAE 1340 steel since its toughness is known to be especially sensitive to strength level. The results obtained on this material were compared with those obtained on SAE 4340 steel which is generally considered as one of the most satisfactory steels at high hardness levels.

Material and Procedure

The SAE 1340 and 4340 steels used in this investigation were obtained as $\frac{3}{4}$-in. and $1\frac{1}{4}$-in. diameter rods, respectively, in the hot-rolled condition. Test specimens were prepared from the rods in the following manner:

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* Presented at the Sixty-first Annual Meeting of the Society, June 22–27, 1958.

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[2] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 952.

1. Normalize at 1675 F for ½ hr and air cool.

2. Stress relieve at 1200 F for 4 hr followed by a furnace cool.

3. Rough machine to one of the shapes shown in Fig. 1. (The SAE 4340 rods were sawed into quarter-sections for the preparation of specimen types 1(*a*) and 1(*c*).)

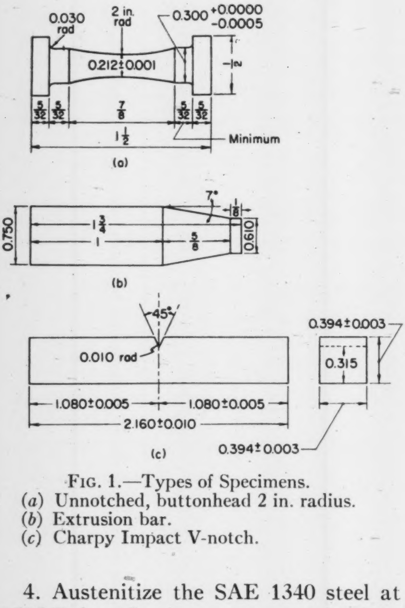

FIG. 1.—Types of Specimens.
(*a*) Unnotched, buttonhead 2 in. radius.
(*b*) Extrusion bar.
(*c*) Charpy Impact V-notch.

4. Austenitize the SAE 1340 steel at 1525 F for 45 min, water quench. Austenitize the SAE 4340 steel at 1550 F for 45 min, oil quench.

5. Temper for 1 hr at 700 F and air cool.

6. Finish machine to the specimen shapes shown in Fig. 1.

The tempering temperature of 700 F was selected as optimum for the investigation since it was low enough to develop a rather high hardness in the steel (Rockwell hardness C 46.6; 237,000 psi tensile strength for SAE 1340), and, on heating for warm working, the steel softened sufficiently so that it could be readily worked at this temperature. Furthermore, strain aging at 700 F previously had been shown to overage the steel. This maximum aging temperature, consequently, would allow studies of both under- and overaged metal. The room temperature tensile ductility at this tempering temperature was also quite high (see Fig. 4).

The mildly contoured specimen, Fig. 1(*a*), was used to determine the unnotched tensile properties of the steel. Previous investigations have shown that the mechanical properties obtained on these contoured specimens were within a few per cent of those obtained on cylindrical bars (**8**).

All tension tests and prestretching operations were conducted with the aid of a specially designed fixture which yielded an eccentricity of less than 0.001 in. This equipment and the testing procedure have been previously described (**9**). Speed of tension testing was made to correspond to a rate of travel of the testing machine head of approximately 0.05 in. per min. Both prestrain and final ductility measurements were made by determining the specimen diameters on a microcomparator before and after each straining operation.

A 3½ ± ½-hr interval was used between prestretching at room temperature and final testing. Specimens prestrained above room temperature were held in the aging furnace for 1 hr followed by an air cool. During the other 2 hr, the specimens were measured and held at room temperature.

Cylindrical bars of the type shown in Fig. 1(*b*) were machined from the SAE 1340 and 4340 steels. These rods were prestrained ($\epsilon_p = 0.41$) by extruding through a 0.610-in. diameter, 7-deg half-angle die at 500 and 700 F.

The extruded specimen blanks were

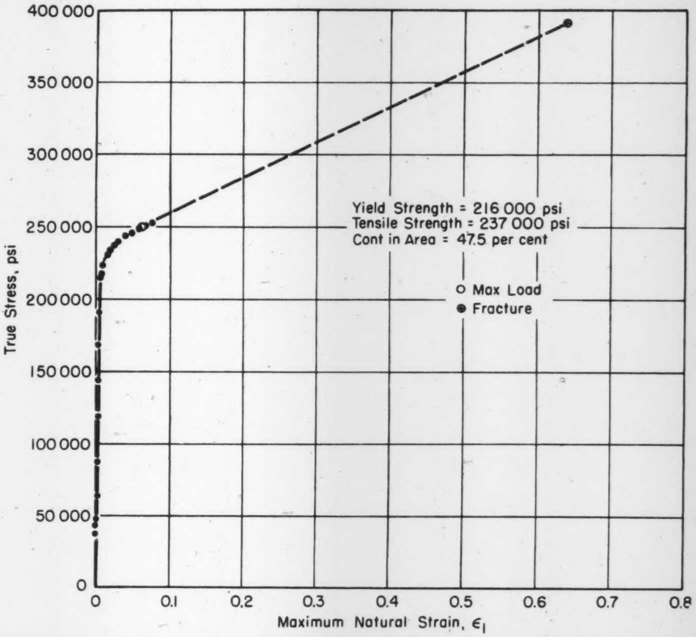

FIG. 2.—True Stress-Strain Curve for SAE 1340 Steel Water-Quenched and Tempered at 700 F.

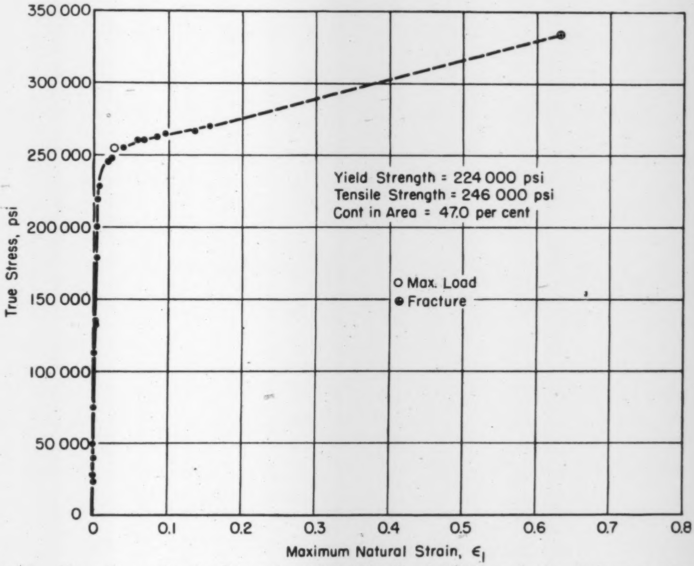

FIG. 3.—True Stress-Strain Curve for SAE 4340 Steel Oil-Quenched and Tempered at 700 F.

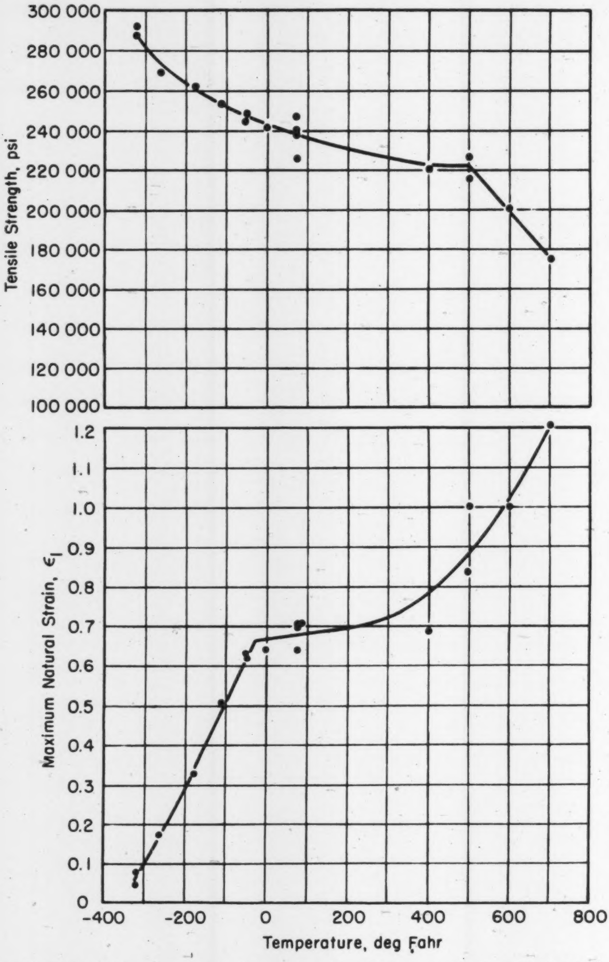

FIG. 4.—Tensile Properties as a Function of Testing Temperature for SAE 1340 Steel Water-Quenched and Tempered at 700 F.

aged for approximately 28 hr at room temperature between prestraining and testing.

RESULTS AND DISCUSSION

The true stress - natural strain curves for SAE 1340 and 4340 steels, quenched and tempered at 700 F, are shown in Figs. 2 and 3.

The manner in which the unnotched tensile strength and ductility of the two steels varied with testing temperature is shown in Figs. 4 and 5. Because the SAE 4340 steel used in this study did not exhibit an unnotched transition above the temperature of boiling nitrogen until the tempering temperature was decreased to 300 F, unnotched tests on this material were discontinued.[3]

Specimens of SAE 1340 prestretched at room temperature, 500 F, and 700 F were tested in tension at two sub-transition temperatures: -260 F, Figs. 6 and 7; and -321 F, Figs. 8 and 9. The low temperature tensile properties after warm stretching were similar to those previously obtained on test specimens of the same material that were prestretched at room temperature and subsequently aged (6). That is, at any constant magnitude of prestrain, the low-temperature strength went through a maximum on 500 F prestraining. Overaging, by stretching at 700 F, decreased the strength and increased the ductility over the values obtained on 500 F prestraining.

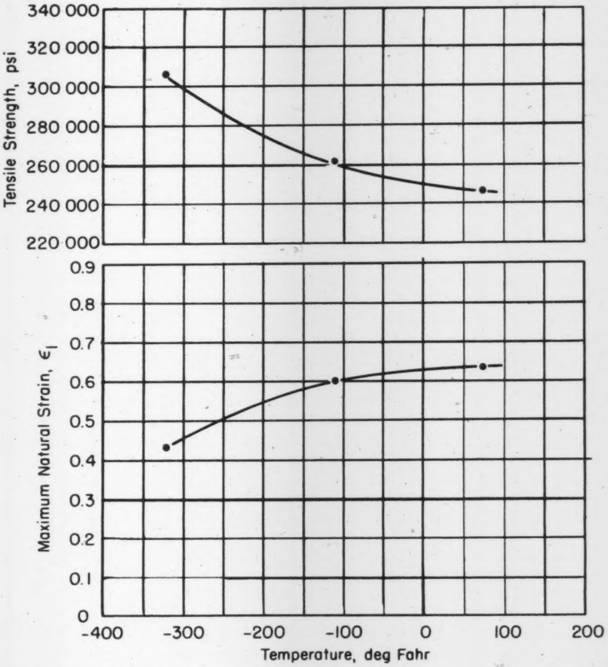

FIG. 5.—Tensile Properties as a Function of Testing Temperature for SAE 4340 Steel Oil-Quenched and Tempered at 700 F.

Since prestrains of $\epsilon_p = 0.40$ were always sufficiently large to effect a complete rheotropic recovery (that is, as represented by the linear portion of the ductility-prestrain curve in Figs. 6 and 8) this magnitude of prestrain was selected for the extrusion portion of this study.

The true stress-strain curve for material extruded at 700 F is shown in Fig. 10. A comparison of the room temperature tensile properties before and

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[3] The maximum temperature at which the metal can be warm-worked is the temperature at which it was tempered. The temperature range between ambient and 300 F was thought to be too narrow to be of interest.

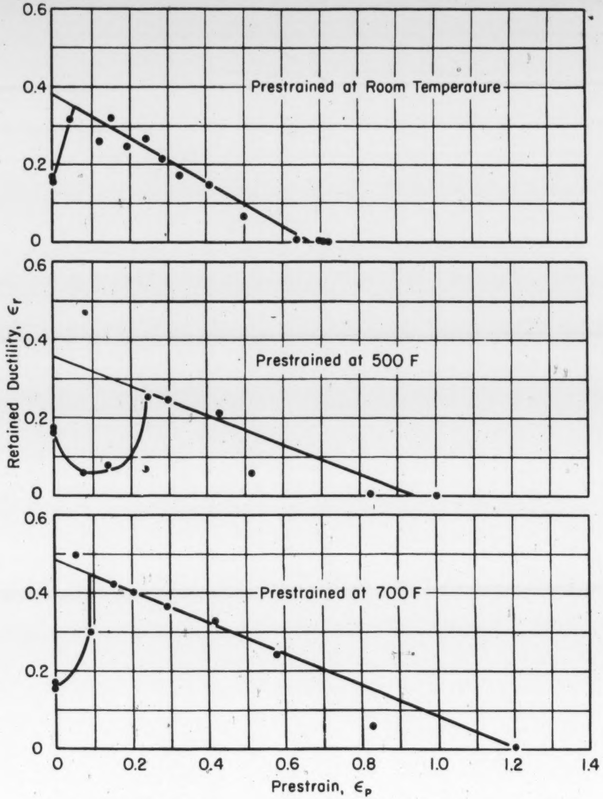

FIG. 6.—Rheotropic Recovery Obtained on SAE 1340 Steel (Water-Quenched and Tempered at 700 F) Prestrained at Indicated Temperatures Followed by Testing at −260 F.

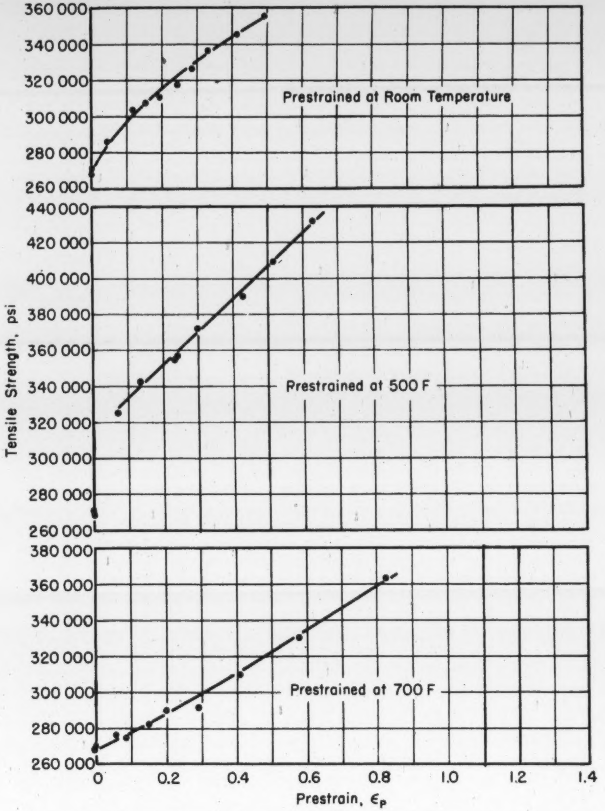

FIG. 7.—Tensile Strength of SAE 1340 Steel (Water-Quenched and Tempered at 700 F) at − 260 F After Prestraining by Stretching at Indicated Temperatures.

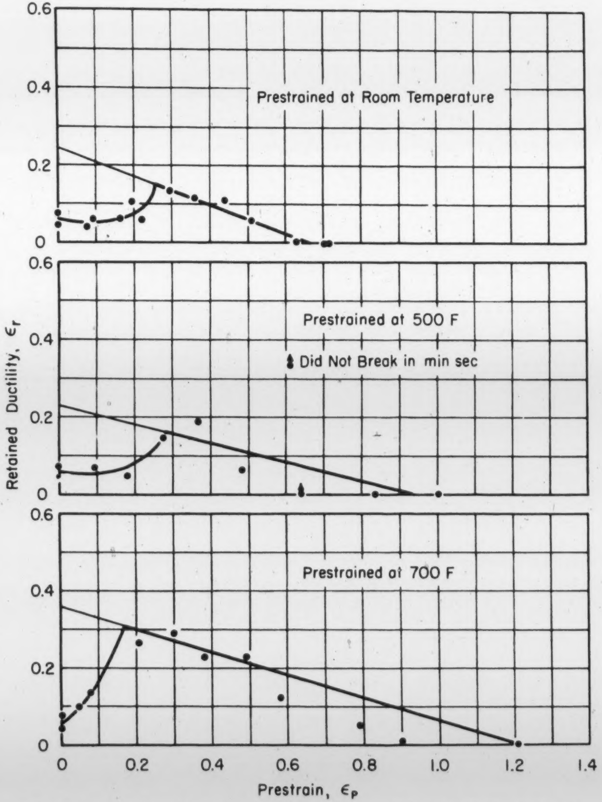

FIG. 8.—Rheotropic Recovery Obtained on SAE 1340 Steel (Water-Quenched and Tempered at 700 F) Prestrained at Indicated Temperatures Followed by Testing at −321 F.

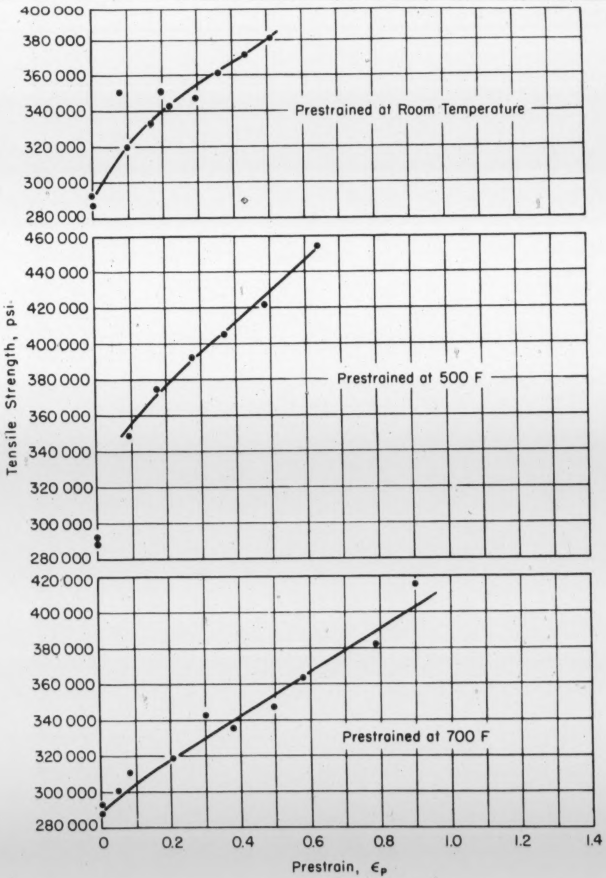

FIG. 9.—Tensile Strength of SAE 1340 Steel (Water-Quenched and Tempered at 700 F) at −321 F After Prestraining by Stretching at Indicated Temperatures.

after extrusion (see Figs. 2 and 10) shows that the extrusion operation raised the yield strength appreciably (from 216,000 to 234,000 psi), while the tensile strength and ductility were increased slightly (from 237,000 to 238,000 psi for the former and from 47.5 to 49.5 per cent reduction of area for the latter). In addition, the strain hardening exponent of the extruded material was lowered so that the fracture stress was decreased (from 392,000 to 340,000 psi).

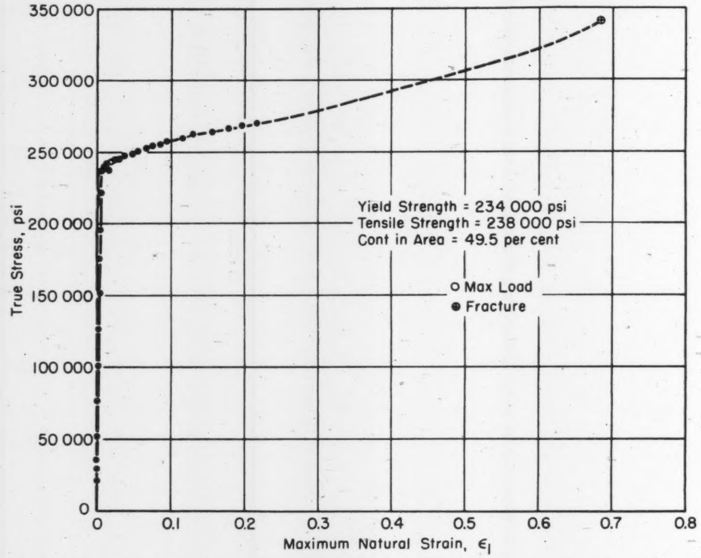

FIG. 10.—True Stress-Strain Curve for SAE 1340 Steel (Water-Quenched and Tempered at 700 F) After Prestraining by Extruding ($\Sigma_p = 0.41$) at 700 F.

Transition curves obtained with Charpy V-notch specimens of SAE 1340 steel extruded at 500 and 700 F are shown in Fig. 11. Data obtained on material in the "as-heat treated" condition are also included for comparison. Rockwell hardness, C-Scale, tests were made on all test bars; these values are shown in parentheses beside each data point. Extruding at either 500 or 700 F lowered the transition temperature to slightly below ambient. The higher temperature extrusion also produced a very high level of toughness at both sub- and super-transition temperatures, while extruding at 500 F did not develop an especially high toughness at super-transition temperatures. With the as-heat treated condition as a basis, extruding at 700 F caused a hardness drop of approximately two points Rockwell hardness, C Scale, while extruding at 500 F increased the hardness by about two points. The lower temperature extrusion, in addition to increasing the hardness a few points, elevated the room temperature toughness (energy absorbed, ft-lb) about threefold. The higher temperature extrusion, while necessitating a mild sacrifice in hardness, yielded a sevenfold increase in the toughness value at room temperature.

Unfortunately, toughness data as a

function of tempering temperature were not available on this heat of SAE 1340 steel so that the loss in hardness resulting from elevated temperature extrusion (from Rockwell hardness C 46.6 to C 44.4) could not be directly evaluated in terms of expected toughness improvement. Baeyertz, Craig, and Sheehan (10), however, have presented Charpy impact *versus* testing temperature curves for a commercial SAE 1340 steel, quenched and tempered over a range of temperatures (Fig. 12). Their heat of steel softened with tempering temperature at a faster rate than the steel of the same nominal composition used herein in that a Rockwell hardness of C 45 was developed on tempering at 700 F as compared with 46.6 obtained with the present heat. Furthermore, their steel was a "higher quality" heat since tempering to a Rockwell hardness of C 49 yielded a higher toughness over the complete range of testing temperatures than the present steel developed when tempered to a Rockwell hardness of C 46.6. Nevertheless, the toughness of the extruded metal with a hardness of just less than Rockwell hardness C 45 was considerably improved over that obtained on the "higher quality" heat of steel when tempered to a Rockwell hardness of C 45. The transition temperature of the extruded metal (as measured by the position of the knee of the curve) was almost identical with that obtained by Baeyertz, Craig, and Sheehan (10) after tempering at 800 F.

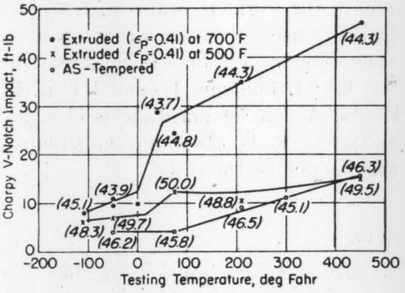

FIG. 11.—A Comparison of Toughness and Hardness of Warm-Extruded and Unworked SAE 1340 Steel (Water-Quenched and Tempered at 700 F), Rockwell Hardness C Values in Parentheses.

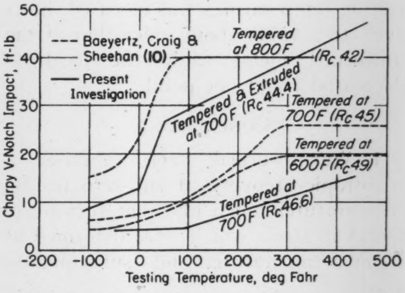

FIG. 12.—Comparison of Toughness Obtained in This Study with That Previously Presented byBaeyertz, Craig, and Sheehan for SAE 1340 Steel (10).

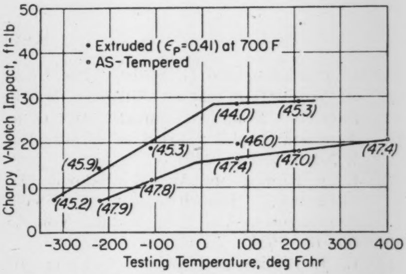

FIG. 13.—A Comparison of Toughness and Hardness of Warm-Extruded and Unworked SAE 4340 Steel (Oil-Quenched and Tempered at 700 F), Rockwell Hardness C Values in Parentheses.

In order to compare the effects of warm-worked SAE 1340, a steel normally considered poor at high-strength levels, with a steel which is more insensitive to strength levels, SAE 4340, Charpy impact tests were made on the latter steel in both the "as-heat treated" (tempered at 700 F) condition and in the heat-treated and extruded (at 700 F) condition (Fig. 13). Unlike the SAE 1340 steel, the SAE 4340 steel did not appear to have its transition temperature lowered (again as indicated by the position of the knee of the curve) by warm deformation. The toughness level at all testing temperatures was elevated, however, to a value about twice that of the unworked metal while the hardness decreased about one point.

CONCLUSIONS

1. Warm-worked steels exhibited a rheotropic recovery at sub-zero testing temperatures much like that previously found in steels which were deformed at room temperature and subsequently aged.

2. An enormous toughness improvement accompanied by an appreciable increase in yield strength, a slight increase in tensile strength and ductility as well as a mild loss in hardness was developed in SAE 1340 steel by warm extruding after heat treating.

3. A much lower toughness improvement was obtained with warm-worked SAE 4340 than with SAE 1340 steel.

Acknowledgments:

This investigation was conducted in cooperation with the Office of Naval Research, U. S. Navy, to whom the author is grateful for permission to publish the work. It is also with a good deal of pleasure that the author acknowledges the many kind suggestions of Mr. W. M. Baldwin, Jr. and Mr. E. B. Evans. A debt of gratitude is also due to Messrs. R. G. Howe and G. Condzer who conducted the tests.

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DISCUSSION

Mr. Stuart V. Arnold.[1]—I should like to inquire what the temperature of the extruded material was as it left the die, assuming that an increase was realized from the energy expended in deformation.

Mr. Eric Kula.[2]—In line with the previous question, did the extra heating for the extrusion process influence the results? In other words, how much of the change in properties could be attributed to changes in structure brought about by working and how much to additional tempering during the time the material was held at the extrusion temperature?

Chairman W. T. Lankford.[3]—I would like to ask the author two questions:

Were any significant differences noted in the composition of the steel used and that reported on by Baeyertz and coworkers (see reference (10) of the paper)?

Also, I wonder whether the author would care to comment any further on the mechanism by which the ductility and toughness were improved by this warm working treatment. By that, I mean any comments you might care to make about the basic metallurgical processes that are taking place.

Mr. C. Mylonas.[4]—Has the author any data on prestraining in a different direction from that of the final testing? Are the results different if the prestraining is done at right angles to the direction of final testing?

In mild steels it appears that prestraining raises the transition temperature. In all of the author's tests it was found that it lowers it and also increases the impact energy appreciably. Is that only due to the temperature of prestraining, since the results I refer to were all reached with mild steel prestrained at room temperature? Do you then think that the high temperature prestraining helps to actually lower the transition temperature, and to reverse the effect completely from what was observed in mild steels prestrained at room temperature?

Mr. E. J. Ripling (*author*).—The interest shown by the discussers in this paper is gratifying.

Messrs. Arnold and Kula are both concerned with property changes that might have resulted either from holding the specimens at the extrusion (or tempering) temperature for some additional time or from a temperature increase that "was realized from the energy expended in deformation." So far as the effect of additional time at the tempering temperature is concerned, the author had shown in an earlier report[5] that retempering 1340 for one hour did not measurably change the properties obtained by an initial one hour temper. In reply to the question of temperature increase produced by the deformation, it is felt that this also is negligible since the extrusion was conducted in a tensile machine at a strain rate of the same order as that used for tensile testing.

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[3] Chief Research Engineer, Specialty Products, United States Steel Corp., Pittsburgh, Pa.

[4] Associate Professor of Engineering, Brown University, Providence, R. I.

[5] D. H. Fisher, R. L. Carlson, and W. T. Lankford, "The Effects of Strain Rate and Temper Rolling on the Strain-Aging Characteristics of Rimmed Deep-Drawing Steel," p. 932, of this publication.

Mr. Mylonas' question on the behavior of specimens which were prestrained in one direction and tested in a second direction can be answered by considering reference (4) of the paper in which specimens were prestrained in compression and tested in tension. This complete reversal of the straining direction produced results similar to those described in this paper.

The second question raised by Mr. Mylonas concerns the effect of prestraining on the transition temperature of mild steel. The apparent contradiction that this discusser points out in the results presented in this paper, that is, a lowering of the transition temperature by working as compared with the results secured by other investigators who found embrittlement to be promoted by cold working, is primarily a matter of the magnitude of the prestrain. Notice in Figs. 6 and 8 that ductility is recovered only after the prestraining has exceeded some critical magnitude.

Although the amount of prestrain that is the critical amount may be a function of prestraining temperature, the low-temperature ductility after prestraining at any temperature can be decreased or increased depending on the prestrain magnitude.

In reply to Mr. Lankford's question on a comparison of this steel composition with that used by Baeyertz and coworkers, these data are not available.

So far as a mechanism to explain rheotropic brittleness, the author, at present, can suggest nothing other than to state that it is obviously some type of strain aging phenomenon. Attempts have been made by others to rationalize the behavior on a dislocation model basis.

EFFECT OF SIZE ON THE YIELDING OF MILD STEEL BEAMS*

BY CEDRIC W. RICHARDS[1]

SYNOPSIS

The upper yield point in mild steel is subject not only to wide variations of a random nature but also to systematic variations depending on specimen size. This dependence on size has been shown to exist in a previous paper dealing with the case of simple tension (**1**).[2] In the present investigation the theory is extended to the case of pure bending and supported by experimental evidence.

A statistically designed and carefully controlled experiment is carried out, which provides tests for the significance of a number of variables including size. The precautions taken to avoid unwanted variables and the actual conduct of the experiment are described in detail.

An analysis of variance is made for the observed experimental results, with the conclusion that size is the only significant effect present. Further study reveals a substantially linear relationship between the logarithms of size and yield strength. This is in agreement with the statistical theory and provides further support for the brittle fracture analogy for the yielding of mild steel.

The sudden breakdown of elastic strength occurring in mild steel when it reaches its upper yield point bears a striking resemblance to the fracture of a brittle material (1). Like the breaking strength of a brittle material, the upper yield point varies widely from specimen to specimen—much more so than does the yield strength of other ductile metals. Like the strength of a brittle material, part of the wide variation in the upper yield point is due to its sensitivity to differences in test conditions such as eccentricity of loading, uniformity of material, and transient effects. Of greater interest, however, is an inherent variation that is a characteristic of the material. This inherent variation is the result of imperfections randomly distributed throughout the material, influencing its strength in the same way that the strength of a brittle material is influenced. The effects of such a random distribution are threefold: (*a*) a statistical variation of strength; (*b*) a strength in bending somewhat higher than that in tension; and (*c*) a marked size effect. These effects, being inherent in the makeup of the material involved, are present even if testing conditions are under complete control. On the other hand, they may be masked by variations caused by uncontrolled conditions.

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* Presented at the Sixty-first Annual Meeting of the Society, June 22–27, 1958.

[1] Assistant Professor, Civil Engineering, Stanford University, School of Engineering, Stanford, Calif.

[2] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 966.

The confused situation that has existed in the investigations of the "raised yield point in bending" over the past twenty-five years appears to be a result of failure to recognize the causes of variability just described. There has, in particular, been an almost complete disregard for the effects of specimen sizes on results. Specimens varying in dimensions by factors as great as 15 to 1 have been used by different investigators. Such size variations alone could account for all of the changes attributed to the "raised yield point in bending."

The purpose of the experimental part of this investigation is to study the variation of the bending yield stress with size, all other factors being placed under careful experimental and statistical controls. From such an investigation, a clear picture can be obtained and a comparison made with the statistical theory.

The results obtained indicate a significant effect of specimen size approximately in accordance with the theory. The absence of other significant factors in the statistical analysis indicates that the experimental controls were adequate.

STATISTICAL THEORY OF YIELDING

The mechanical behavior of mild steel may be represented by the following model: A material that is made up of two components, (*a*) a strong, brittle component, and (*b*) a soft, easily deformable component. The latter is supported in some way by the former, making possible elastic behavior at high stresses. At a certain stress, the brittle component fractures and the entire stress is thrown onto the soft component, the material thereafter behaving in a ductile manner. The stress at which the brittle component fractures is the upper yield point. Consequently the beginning of yielding at the upper yield point may be expected to follow the same fundamental laws as the fracture of brittle materials. This is the basis for the analogy between the beginning of yielding in mild steel and brittle fracture.

The theory of the strength of brittle materials has been developed by Weibull (2) and extended to cover several states of stress, including tension and bending. His theory for simple tension has been adapted directly to the beginning of yielding in tension (3). In the case of bending there is an important difference between brittle fracture and the beginning of yielding. Because of the marked weakness of brittle materials in tension, as contrasted with their strength in compression, failure in bending always occurs on the tension side of the beam. Only half of the beam is therefore considered in the derivation of the theory for brittle fracture. Yielding, on the other hand, can begin with equal ease in compression or tension. It follows that the derivation of the theory for the beginning of yielding must include the entire volume of the beam. Pure bending is chosen for this derivation, since it represents a simple one-dimensional stress distribution and has been used by most other investigators.

In pure bending, all fibers at equal distances from the neutral surface are subject to the same strain. It is assumed that yielding occurs in tension and compression under identical conditions. It is further assumed that a volume element extending the length and width of the beam and having an infinitesimal thickness will be governed by the same rules as an element of a specimen in simple tension or compression. The probability $P(\sigma)$ that yielding will begin at or below a stress σ in such an element is given by Eq 1 (2, 3):

$$d[\log (1 - P)] = -n(\sigma)\, dv \ldots\ldots (1)$$

Here $n(\sigma)$ is a material function representing the statistical distribution of strengths from element to element in the material. The expression $n(\sigma)$ may also be thought of in terms of the number of flaws having a critical stress σ per unit of volume of the material. Taking $n(\sigma)$ to be of the form $k\sigma^m$ and noting that $dv = bldy$, and $\sigma = \sigma_1 \cdot \frac{y}{h}$ (where σ_1 is the extreme fiber stress and the depth of the beam is $2h$), Eq 1 becomes

$$d[\log(1 - P)] = -blk \left(\sigma_1 \frac{y}{h}\right)^m dy$$

Integrating this expression over the entire depth of the beam we find the probability of yielding for any given beam

$$P(\sigma_1) = 1 - e^{-k\sigma_1{}^m v/m+1}$$

From this probability distribution function the mean value of σ_1 for yielding of a given steel may be computed,

$$\overline{\sigma_1} = \int_{\sigma_1=0}^{\sigma_1=\infty} \sigma_1 \, dP = \int_0^\infty e^{-(k\sigma_1{}^m v/m+1)} \, d\sigma_1$$

or

$$\overline{\sigma_1} = \frac{I_m(m+1)^{1/m}}{k^{1/m}} \cdot \frac{1}{v^{1/m}} \ldots\ldots (2)$$

in which k and m are material constants,

$$I_m = \int_0^\infty e^{-z^m} \, dZ,$$

v = volume in pure bending, and σ_1 = extreme fiber stress.

Equation 2 has two important implications. First, the yield stress in bending varies with the specimen size in exactly the same way as does the tensile yield stress (1). Second, comparing with the corresponding expression for the mean tensile yield stress

$$\overline{\sigma_y} = \frac{I_m}{k^{1/m}} \cdot \frac{1}{v^{1/m}} .$$

it may be seen that for specimens of equal volumes

$$\bar{\sigma}_1 = (m+1)^{1/m} \cdot \bar{\sigma}_y$$

This shows the effect of the nonuniform stress distribution and may partially explain the "raised yield point in bending."

Equation 2 may be written

$$\overline{\sigma_1} = \frac{A}{v^{1/m}}$$

where A and m are material constants. Taking the logarithms of both sides

$$\log \overline{\sigma_1} = \alpha - \frac{1}{m} \log v \ldots\ldots\ldots (3)$$

$$S = \alpha + V \ldots\ldots\ldots\ldots (3a)$$

where $S = \log \overline{\sigma_1}$, α is a material constant and $V = -\frac{1}{m} \log v$. Equations 3 and 3(a) will be used to represent the theory of yielding since they are linear in the logarithms of the two variables.

DESIGN OF EXPERIMENT

The primary purpose of this investigation is to study the relationship between *yield strength* and *specimen size* for the case of pure bending. In setting up and conducting the experiments, therefore, every effort was made to minimize other variables. Precautions taken include the use of a single source of materials, heat treatments to insure uniformity, dimensional similarity of specimens, accurate load application, and careful control of environment and testing procedures. Many variables are however difficult, if not impossible, to reduce to negligible values. Consequently statistical controls were used extensively in addition to the above physical precautions.

The material selected for these experiments was a commercial quality killed structural mild steel, chosen as a type that could be expected to have a pronounced upper yield point. It was obtained directly from the steel mill in the form of hot-rolled round bars, $1\frac{1}{2}$ in. in diameter and 10 ft long, all rolled from the same heat. The ladle analysis was as follows:

Carbon, per cent	0.18
Manganese, per cent	0.40
Phosphorus, per cent	0.014
Sulfur, per cent	0.30
Silicon, per cent	0.20
Copper, per cent	0.14
Tin, per cent	0.034

Beams of five different sizes were used in the investigation, ten in each size, to provide data for the statistical analysis. Dimensions of the five sizes, series B1 through B5, are given in Table I. It should be noted that all sizes were made dimensionally similar. The ratio between the dimensions of the largest and smallest sizes was 6.3, and intermediate sizes were so arranged that the ratio of adjacent sizes was constant (1.59). In this way the results are equally spaced on the scale representing log *v* (*see* the last column in Table I).

TABLE I.—BEAM DIMENSIONS.

Series (B*j*)	Width, *b*, in.	Depth, *h*, in.	Span, *g*, in.	Moment Arm, *e*, in.	Roller Diam., *d*, in.	$\log_{10} 10h$
B1....	0.0635	0.1585	1.840	0.460	0.161	0.2
B2....	0.1005	0.251	2.912	0.728	0.250	0.4
B3....	0.1590	0.398	4.615	1.154	0.391	0.6
B4....	0.2525	0.631	7.32	1.83	0.625	0.8
B5....	0.4000	1.000	11.60	2.90	1.000	1.0

NOTE. $b = 0.4h$ $e = \frac{1}{4}g = 2.9h$
$g = 11.6h$ $d \approx h$

The loading arrangement used to produce pure bending is shown in Fig. 1. Hardened steel rollers were used both for supports and for applying the loads in order to eliminate axial restraining effects. The dimensional similarity between the beams of different sizes was extended to include the supporting rollers in order to insure that the contact stresses would be the same for all sizes (*see* Appendix I). The rollers were held in place by specially made jigs while the initial load was applied. The jigs were designed to hold the rollers parallel and at the correct spacing to the nearest 0.001 in. After application of the initial load, clips holding the rollers against the jig were released and the jig could be removed by sliding it away from the beam.

The actual source of each specimen in the original stock was determined by statistical considerations. It was felt that there might well be significant variations in the material as it came from the source, from one bar to another, and from point to point in each bar. To test for the significance of these variations and place them under proper control a statistical design was developed. In this design the factors to be controlled were sizes, positions, and bars. The statistical model can be written as a generalization of Eq 3(*a*):

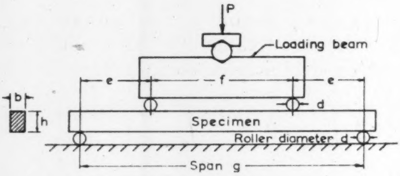

FIG. 1.—Pure Bending Loading Arrangement.

$$\text{Expected value of } S_{ij} = \alpha + V_j + P_k + B_i \quad (3b)$$

where:

S_{ij} = the logarithm of the strength,
V_j = the effect due to size,
P_k = the effect due to position,
B_i = the effect due to bars,
i = Number of stock bar (1, 2, 3, 4, $\cdots$ 10), and
j = Series number designating size (1, 2, 3, 4, 5).

The original 10-ft bars were sawed into 5-ft lengths and arranged in a random order with all sheared ends on one side and sawed ends on the other. Each bar was then divided into five 1-ft segments, and the segments were numbered according to a latin square. Figure 2 shows the latin square for the first five bars. The same arrangement was used for the second five bars. Specimens were machined from the centers of the 1-ft segments. Each of the main effects, sizes, positions and bars appeared at five levels in each square. Each bar contained a complete set of specimens of the five sizes used, and each size included specimens from all ten bars.

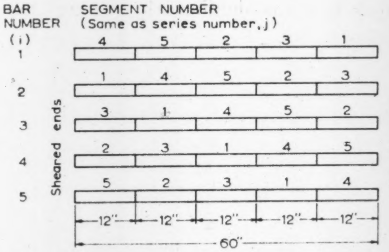

FIG. 2.—Latin Square for First Five Bars.

In addition to the statistical controls, the uniformity of the material was improved by suitable heat treatments. After all of the 1-ft segments had been cut and marked, they were given a preliminary homogenizing anneal at 1650 F. After machining, the specimens were given a stress relief anneal at 1100 F in a salt bath, intended to remove the effects of machining, if any (details are given in Appendix II). Considerable care was exercised in the machining process to avoid heavy cuts and overheating.

Environment and testing procedures were also carefully regulated. Near the upper yield point the condition of mild steel is highly unstable. If the loading apparatus stores large amounts of strain energy, transient disturbances can release it into the specimen causing premature yielding. This was avoided by using a very rigid machine and taking precautions against shocks and vibrations in the immediate vicinity. A 120,000-lb hydraulic testing machine was used because of its high stiffness. The loading beams were designed to be as rigid as possible within the limits of practicability. Including all connected apparatus (testing machine, weighing capsule, and loading beam) the stiffness measured approximately 500,000 lb per in.

Strain rate was held constant at all times during the loading of the specimens. It was set at 15×10^{-6} per sec, which is slow enough to be on the flat part of the curve of yield strength *versus* strain rate for mild steel (**4**). Thus small variations in strain rate would not be expected to have any noticeable effect.

Each day's tests were arranged to include a complete set of specimens from one stock bar, representing all five sizes. In this way variations that might exist from day to day could not be attributed to size.

In order to check the effectiveness of the experimental controls, a large number of auxiliary tests were made. These included microstructure examinations, hardness tests, additional bending tests, and tests to determine the creep in the SR-4 gages and the effectiveness of the pure bending setups.

Metallurgical examinations were made at all stages in the processing of the steel. They indicated that all microstructures were the same. At a magnification of 500× no signs of deformation of the pearlite or of decarburization could be seen. The carbon content was judged to be the same in all samples.

Hardness tests, also made at various stages in the processing, revealed no significant nonuniformity in the material. Since hardness tests always involve considerable amounts of plastic flow, they do not provide any direct indication of the yield strength but were used only as a further check on the uniformity of the material. Macro- and microhardness tests made on the homogenized stock and on the final beams showed only slight variations, and those of a random nature. Comparisons of beams of different sizes indicated that any variations in hardness that did exist across their cross-sections were of approximately the same magnitude in all sizes.

The auxiliary bending tests provide direct indications of the importance of several possible variables with respect to the yield strength. Ten beams of each of two sizes were tested in a manner identical to that used in the main tests. The large size (BS4) was the same as series B5 and the small size (BS3) midway between B2 and B3 (approximately $\frac{5}{16}$ in. in depth). Half of the specimens of each size had surface layers removed by etching to a depth of 0.001 to 0.003 in. on the extreme surfaces. It was felt that if the surface layers retained any effects of machining after the stress relieving heat treatment this would tend to remove these effects. No significant variations in the yield strengths could be detected. In some cases, the etched beams gave lower values and in others higher values than those for the unetched beams.

Beams taken from either end of the stock bar were compared. In one case a significant variation was noted, the beams taken from the sheared end being noticeably stronger than those from the sawed end. In the other case there was no significant change. As noted previously, the experiment was designed statistically to give proper consideration to any such variation in other stock bars.

The most interesting variation was one that was discovered in the case of small beams taken from different parts of the cross-section of the stock bar. Two of the ten small size (BS3) auxiliary beams were machined from the center of the $1\frac{1}{2}$-in. round stock as were all the specimens in the five main series; four were from points $\frac{3}{8}$ in. from the center; and four were from the outer surface. Locations are shown in Fig. 3. The yield strengths of the beams from these three locations were found to differ significantly. The figures adjacent to the positions show the average yield strengths in kips per square inch. The number of specimens involved in each case was rather small to provide conclusive evidence, but the results indicate that the material at the center of this particular stock bar was significantly weaker than that nearer the surface. Since the beams used in the main tests were all taken from the center of the stock bar, the extreme fibers of the smallest beams were closest to the center. A decrease in yield strength toward the center would therefore produce a corresponding decrease in yield strength with size—an effect just opposite that predicted by the theory.

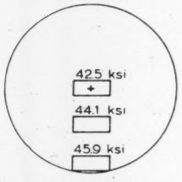

Fig. 3.—Locations of BS3 Beams in Cross-Section of Stock Bar.

Experimental Procedure

In accordance with the statistical design, the order in which specimens of each day's set were tested was determined by lot and was unrelated to the order of the specimens in the bar.

Basically, the procedure consisted simply of loading each beam until the beginning of yielding was observed. In practice this involves devising a means of detecting the initial yield with the least possible uncertainty—a by no means easy task. Several methods have been used for this purpose by different investigators, with varying success.

One method is the direct observation of yielding itself as evidenced by Lüder's lines. In the case of small beams this is fairly clear cut, but in the larger ones some difficulty is found in detecting the very first slip band as soon as it forms.

Another method involves plotting load *versus* deflection or strain and observing the point at which there is a deviation from linearity. With the highly sensitive strain gages now available, this becomes quite practical and is the principal method relied upon in these experiments.

A third method depends on the observation of permanent set upon unloading after yielding has started. This method provides an indication at the time of the test, whereas the deviation from linearity can be seen only after some calculations or the plotting of a graph. This is also a practical method with the use of sensitive strain gages.

A combination of the last two methods was used in all tests in this investigation. Strains were measured with SR-4 electric resistance wire strain gages attached to the tension surface of each beam. Since the beams were loaded in pure bending, the maximum fiber stress was constant over the length of the center span. Yielding itself is highly localized and may occur at any point along this span. In order to be sure to detect the first yielding, therefore, the strain gages were selected to cover as nearly as possible the entire length subject to pure bending in each beam. Thus average strains were measured, as is usually the case in engineering tests.

A standardized testing procedure was set up and carefully followed in all tests. A small initial load, roughly one third of that corresponding to the lower yield point in tension, was applied and the jig was removed. The load was then cycled several times between the initial load and a "zero" load, which was approximately two thirds of the lower yield point load. This was for the purpose of lessening the effects of creep in the strain gages. Preliminary tests using a cold-rolled steel beam well within its elastic range revealed a certain amount of creep in the gages. When the load was cycled a number of times, the strain gage readings at the lower load gradually increased at first and settled down to a fairly constant value only after several cycles. Similar behavior in the mild steel beams was therefore also attributed to creep in the gages.

After the strain reading had become stabilized, the load was increased at the predetermined rate and simultaneous readings of load and strain were recorded at regular intervals. When the load reached a value near the lower yield point load, it was reduced again to the zero load in order to detect the appearance of permanent set, if any. The load was then increased to its highest previous value without any readings being taken, and further readings at higher loads were recorded. This cycle was repeated several times until yielding was clearly evident. The result was a frequent check for permanent set during the test and a continuous series of load *versus* strain readings. In the smaller beams, yielding was quite abrupt, even accompanied by a drop in the load in many cases.

It should be mentioned that considerable difficulty was encountered in connection with the smallest beams (series B1). In the first place, no standard strain gages could be found that would fit on their $\frac{1}{16}$-in. wide faces. And although very carefully made, the special gages that were used were found to be erratic in their behavior because of their small size. In the second place, the problem of loading without eccentricity increased rapidly with decreasing size, and some unavoidable eccentricity was noticeable in this smallest size. These difficulties, combined with the possibility of pronounced weakness in the material itself due to its source in the center of the stock bar, tend to cast some doubts on the validity of the results of series B1.

TEST RESULTS

The load at which the first departure from linearity occurred was determined by the following method. Using all readings up to a point just below what appeared to be the yield load a straight line of the form

$$\epsilon = a_0 + a_1 P$$

was fitted by the method of averages. Using this equation, the values of

$$\delta = \epsilon_{\text{obs}} - \epsilon$$

were then tabulated, where ϵ_{obs} is the observed strain at any given load and δ is the deviation of this strain from the linear relationship assumed. Continuing this tabulation beyond the linear portion of the curve, it was possible to see quite distinctly the beginning of yielding. It was found that in general below the yield load the observed strains stayed within 2 or 3 microinches per in. of the calculated values. (Occasional deviations of 5 or 6 were noted, but since they appeared far down in the elastic range it was felt that they must be simply experimental error.) At the yield load, the deviations increased steadily. In all cases it was possible to fix upon a very small deviation as the criterion for the beginning of yielding. For the smallest beams, the value was chosen as 6 microinches per in., with 4 or 5 for the larger beams. This corresponds to an offset of from 0.0004 to 0.0006 per cent.

In five cases, what was felt to be a significant permanent set began to appear at loads that turned out to be slightly lower than those corresponding to the above offsets. In order to be on the conservative side, these lower loads were chosen as representing the beginning of yielding. The permanent set used as a criterion was 10 microinches per in., somewhat larger than the offset to allow for a slight creep in the strain gages.

TABLE II.—OBSERVED YIELD STRESSES.

Series	B 1	B 2	B 3	B 4	B 5
Specimen Number in Each Series	$(\sigma_u)_{ij}$, psi	$(\sigma_u)_{ij}$, psi	$(\sigma_u)_{ij}$, psi	$(\sigma_u)_{ij}$, psi	$(\sigma_u)_{ij}$, psi
1	51 450	48 530	45 010	35 890	36 980
2	44 520	51 760	45 140	41 300	37 720
3	52 470	53 120	39 570	46 220	36 980
4	49 360	47 500	42 590	34 460	36 510
5	46 940	46 910	35 030	30 450	32 815
6	49 270	51 260	44 700	42 930	33 460
7	49 190	49 910	45 800	39 400	36 540
8	52 020	48 430	43 590	31 540	35 180
9	41 600	53 500	46 670	37 930	35 200
10	50 000	51 930	43 570	36 790	33 360

NOTE: $(\sigma_u)_{ij}$ = Maximum stress at yield.

Results were recorded in terms of the following quantities:

v_{ij} = volume of each specimen

$$\bar{v}_j = \frac{\sum_{i=1}^{10} v_{ij}}{10}$$

$x_j = \log_{10} \bar{v}_j$

$(\sigma_u)_{ij}$ = yield stress for each specimen (Table II)

$$(\overline{\sigma_u})_j = \frac{\sum_{i=1}^{10} (\sigma_u)_{ij}}{10}$$

$$y_{ij} = \log_{10} (\sigma_u)_{ij}$$

$$z_j = \overline{y_j} = \frac{\sum_{i=1}^{10} y_{ij}}{10}$$

where i = Number of stock bar (1, 2, 3, 4 ⋯ 10) and j = series number designating size (1, 2, 3, 4, 5). Mean values for the five series are shown in Table III.

Interpretation of Results:

In the study of the results of experimental work there are two important questions:

1. Which effects, if any, are significant?

TABLE III.

Series, (B_j)	Mean Volume, $\bar{v}_j$ cu in.	Log Mean Volume $x_j = \log_{10} \overline{v_j}$	Mean Stress, $(\overline{\sigma_u})_j$, psi	Mean Log Stress, $z_j = \bar{y}_j = \overline{\log_{10} \sigma_u}$
B1......	0.009389	−2.02739	48 680	4.68635
B2......	0.036620	−1.43628	50 280	4.70101
B3......	0.146009	−0.83562	43 170	4.63377
B4......	0.582847	−0.23445	37 690	4.57285
B5......	2.321566	+0.36579	35 470	4.54943

2. Do the significant effects fit the proposed theory?

The first question is answered in part by assuming that efforts to make certain factors negligible by careful experimental procedures have been successful. Such factors include environment and testing techniques such as eccentricity of loading, strain rate, and accuracy of strain measurements.

Another and equally important answer is given through the statistical design. The significance of a number of effects can be tested by an analysis of variance. Such an analysis was made for the two Latin squares comprising the five B series of fifty specimens. The main effects were sizes, positions and bars, reflecting possible variations due to variability of materials and treatments as well as those due to specimen size. Positions and sizes were the same for the two squares, but the experimental material (bars) was different. Equation 3(*b*) represents the model and is repeated here:

$$\text{Expected value of } S_{ij} = \alpha + V_j + P_k + B_i \quad (3b)$$

The effect of volume is further subdivided into

$$V_j = \beta(V_j^1) + \gamma(V_j^1)^2 + \delta(V_j^1)^3 + \phi(V_j^1)^4$$

in order to test for possible terms beyond the linear term predicted by the theory.

The test for significance of the main effects was performed by the following analysis:

1. Let the two Latin squares be called X and Y, and define $W = X + Y$ and $Z = X - Y$.

TABLE IV.—ANALYSIS OF VARIANCE TABLE.

Source	Sums of Squares	Degrees of Freedom	Mean Square
Sizes.............	0.322664	4	0.080666
Positions..........	0.0192294	4	0.004807
Bars.............	0.0432525	8	0.005407
Sizes × squares interaction......	0.0175849	4	0.004396
Positions × squares interaction...........	0.0043848	4	0.001096
Residual..........	0.089795	24	0.003741
Between squares...	0.002603	1	0.002603
Total...........	0.102716	49	

2. Compute the analysis of variance tables for W and Z in the standard manner (for example, see Bowker and Lieberman (5)). Table IV shows the analysis of variance.

3. Tests for significance are based on 5 percentage points of the F distribution, as tabulated in Bowker and Lieberman. The criterion is as follows:

If the ratio

$$F = \frac{MS \text{ (main effect)}}{MS \text{ (residual)}}$$

is less than $F_{0.05,\nu_1\nu_2}$ the hypothesis is accepted that there is no effect. Otherwise it is rejected. The following are the results of this test:

(*a*) Sizes: $F = 21.56$

$$F_{0.05,4,24} = 2.78$$

(*b*) Positions: $F = 1.283$

$$F_{0.05,4,24} = 2.78$$

(*c*) Bars: $F = 1.44$

$$F_{0.05,8,24} = 2.36$$

These tests for significance provide the answer to the first question: Which effects, if any, are significant? From the first test it is seen that a highly significant size effect does exist. The effects of positions and stock bars are shown to be insignificant.

4. A detailed examination of the linear, quadratic, cubic, and quartic effects indicates a strong linear effect plus a smaller, but significant cubic effect. The F-ratios are as tabulated below, compared with $F_{0.05,1,24}$:

Source of Variation	Sum of Squares	Degrees of Freedom	$F_{0.05,1,24}$
Linear Sizes	0.266886	1	$71.4 \gg 4.26$
Quadratic Sizes	0.015114	1	$4.04 < 4.26$
Cubic Sizes	0.039259	1	$10.50 > 4.26$
Quartic Sizes	0.001405	1	$0.38 \ll 4.26$
	0.322664	4	

This suggests that the answer to the second question may be qualified to some extent. The presence of the strong linear effect confirms the theory, but the significant cubic effect indicates that there may be other complications present.

The experimental values of x and z have been plotted in Fig. 4. Both the size effect and the nonlinearity of the variation are evident from this plot. There are two possible interpretations of the nonlinearity. Either the linear relationship predicted by the theory is incorrect, or there are other effects present that account for the deviation. From Fig. 4 it

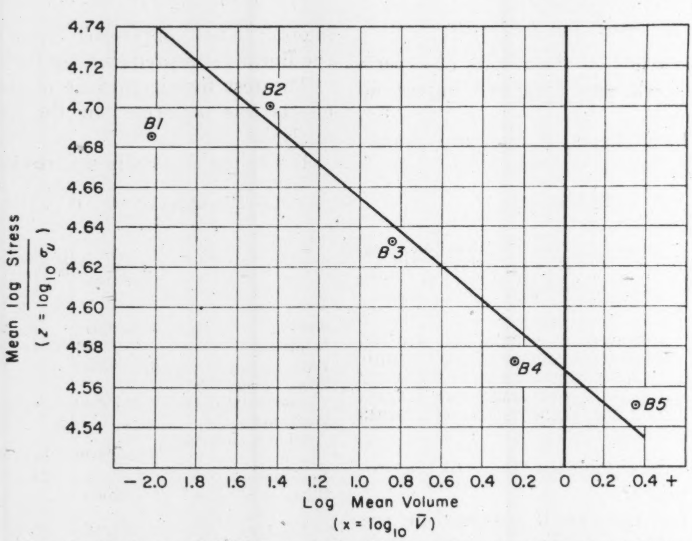

FIG. 4.—Variation of Yield Stress in Bending as a Function of Specimen Size.

appears that the relationship between points B2, B3, B4, and B5 is much more nearly linear than that including point B1. Two previously noted considerations may account for this. First, there was some doubt cast on the validity of the results of series B1 because of the erratic behavior of the strain gages and the difficulty in loading. Second, the size effect may have been noticeably offset in the smallest size because of weakness of the material at the center of the stock bar.

TABLE V.—VALUES OF $\tilde{\sigma}_u$.

Series	Observed $\tilde{\sigma}_u$, psi	Expected Value, $\tilde{\sigma}_u$, psi	Difference, per cent
No. B1..........	48 680	55 240	−11.9
No. B2..........	50 290	49 160	+2.3
No. B3..........	43 170	43 660	−1.1
No. B4..........	37 690	38 770	−2.7
No. B5..........	35 470	34 430	+3.0

With these considerations in mind a regression analysis was made, using only the four larger sizes, series B2, B3, B4 and B5. Since, in the analysis of variance, effects other than size proved to be insignificant, the simple regression analysis provides valid tests of significance when only part of the experimental data is being studied. Accordingly, a straight line was fitted to the plotted points by the method of least squares, giving the following equation for the expected value of z:

$$\tilde{z} = 4.57 - 0.0858x \dots\dots\dots (4)$$

This is the line shown on the graph (Fig. 4). The F-test for goodness of fit is made by comparing the mean square values A and B, where A represents an estimate of the variability of the observations independent of the type of curve assumed, and B represents an estimate of their variability depending on the linear relationship. If the ratio of B to A is greater than $F_{0.05,\nu_1\nu_2}$ we accept the hypothesis that there is no linear relationship. In this case

$$\frac{B}{A} = 1.919 \text{ while } F_{0.05,2,36} = 3.26$$

and a definite linear relationship does exist.

Table V gives for comparison the observed and expected values of $\tilde{\sigma}_u$.

CONCLUSIONS

A real size effect is present in the bending yield strength of mild steel beams.

In the four larger sizes the bending yield strength followed closely the relationship

$$\tilde{\sigma}_u = \frac{A}{v^{1/m}}$$

in which $A = 37{,}000$ psi, $m = 11.7$. This is in accordance with the statistical theory.

Further tests are needed to establish a correlation between yield strength in bending and in tension. Such tests are under way.

Acknowledgment:

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He is indebted to Professor Gerald J. Lieberman and Donald Guthrie of the Applied Mathematics and Statistics Laboratory, for their generous advice and assistance in setting up the statistical controls and in analyzing the results.

He also takes this opportunity to express his thanks to Columbia-Geneva Steel Co., who donated the steel used in these experiments.

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APPENDIX I

The contact stresses between a cylinder and a plane may be calculated by Herz' formula, provided only elastic deformations are involved:

$$\sigma \max = 0.59 \sqrt{\frac{P^1 E}{d}}$$

where P^1 = the load per unit length along the axis of the cylinder, d = the diameter of the cylinder and E = the modulus of elasticity of the material. In order to insure equal contact stresses at yielding in all cases, it is clearly necessary that the ratio of P^1 to d be held constant. The value of P^1 depends upon the load applied at yielding and the width of the beam. If the yield stress were constant for all sizes then the load would depend only on the beam dimensions. Since the actual yield stress is the subject of the investigation and is somewhat unpredictable it is assumed to be constant in the following derivation.

Consider two beams, dimensionally similar, with dimensions in the ratio n. Then $b_2 = nb_1$, $h_2 = nh_1$, $g_2 = ng_1$ and $e_2 = ne_1$ (see Fig. 1). The two section moduli are related as follows:

$$Z_2 = \frac{b_2 h_2^2}{6} = n^3 \cdot \frac{b_1 h_1^2}{6} = n^3 Z_1 \, .$$

For equal maximum bending stress, σ we have

$$M_2 = \sigma Z_2 = n^3 \sigma Z_1 = n^3 M_1$$

where M is the bending moment in the center section and is given by

$$M = \frac{P}{2} e.$$

It follows that

$$\frac{P_2}{2} e_2 = n^3 \frac{P_1}{2} e_1$$

or

$$\frac{P_2}{P_1} = n^3 \frac{e_2}{e_1} = n^2$$

Since each roller takes one half the total load P, and the length of the line of contact is b, the load per unit length becomes

$$P^1 = \frac{P/2}{b}$$

Hence

$$\frac{P_2^1}{P_1^1} = \frac{P_2}{P_1} \cdot \frac{b_1}{b_2} = n$$

Finally, since P^1/d must be constant, we have

$$\frac{d_2}{d_1} = \frac{P_2^1}{P_1^1} = n$$

which means that the roller diameters must be in the same proportion as all other dimensions.

Some degree of approximation is involved, since (*a*) the contact stresses are probably not strictly elastic, and (*b*) the yield stresses are not actually the same for all beams.

APPENDIX II

HEAT TREATMENT DETAILS

	Homogenizing Anneal	Stress Relief
Furnace		
Type	GE Heat Treat Electric (box type)	Electric pot
Dimensions	Hearth 22 by 10 in. arch $7\frac{1}{4}$ in.	4 in. diameter by 13 in. deep
Total weight of iron used	150 lb	Samples only
Salts		50 per cent $NaNO_2$ 50 per cent KNO_3
Annealing temperature	1650 F	1100 F
Time to reach temperature	3 hr	1 to 2 min
Time at temperature	1 hr	25 to 30 min
Time to cool	48 hr	...
Temperature on removal	200 F	350 F
Subsequent treatment		H_2SO_4 pickle, with electrolytic smudge removal

DISCUSSION

MR. R. H. HEYER.[1]—Were possible metallurgical differences through the thickness of the test materials taken into account in preparing the specimens?

MR. C. W. RICHARDS (*author*).—The specimens were all taken from the center of the $1\frac{1}{2}$-in. diameter stock bars. Microstructure examinations were made across the cross-section of the bars, both before and after the homogenizing anneal. Hardness measurements were also taken across the cross-section and other tests were made, as described in the paper.

MR. T. J. DOLAN.[2]—The author's approach appears to be a very interesting and fruitful method of looking at the strength of materials. Certainly there are materials strength variations from crystal to crystal or between different orientations within a single crystal. In the initial portion of the analysis, the author uses a function $n(\sigma)$ as a material function representative of the statistical distribution of strengths from element to element. Was this function meant to cover variations from crystal to crystal or for some other size or type of constituent for the strength considerations of this particular analysis? How does one determine this function or its range of variation? Can one work backwards from observed data on the "size effects" and determine the statistical variation of strength within individual elements from the over-all observation of large scale behavior employing the author's analysis?

In connection with the preparation of the beams themselves, I would like to ask how the surfaces were finished. This might be a vital statistic since we find in fatigue specimens a great deal of variation in fatigue strength depending on how the surface is finished. Minor operations such as polishing with emery paper can leave thin skins of hardened cold-worked material which cannot be detected by, say, a Rockwell hardness test but which might vitally affect fatigue strength. If surface effects of this type were present to the same depth on both the large and small beams it would tend to affect the performance of the small beams by a greater percentage than for the larger depth beams.

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[2] Head, Department of Theoretical and Applied Mechanics, University of Illinois, Urbana, Ill.

MR. RICHARDS.—In answer to the second question, we did everything we could to avoid any hardened skin on the surface. The machining was done very carefully and there was a stress relief anneal following machining. Auxiliary tests are described in the paper, in which the surface layers were etched away on a number of beams. These beams were tested in the same manner as the others and no significant variation between the two types was observed. It was therefore assumed that no skin effect was present.

In answer to the first question, the volume elements were not intended to be necessarily crystals, but the more general kind of element usually used in the study of continuous, isotropic materials. It is recognized that mild steel can only be treated in this way, on a macroscopic scale, but it serves the purpose in this particular case.

I have not tried working backwards to determine the distribution n (σ) but it would be interesting and has been considered.

MR. J. L. M. MORRISON[3] (*by letter*).—In his introductory remarks, the author refers to the confused situation that has existed over the past twenty five years because of, among other things, "an almost complete disregard for the effects of specimen sizes on results." Although this remark is largely justifiable, the author seems to have overlooked work which has been published during and indeed before that period. Thus Cook[4] in 1931 published the results of tests on thick cylinders subjected to internal pressure, which showed clearly that there was an important size effect and that this effect was primarily dependent on the internal diameter of the tube. Again in 1939 the writer[5] published the results of tests in which a comparable effect of size in torsion and in flexure was demonstrated. So far then the author and the writer are in qualitative agreement. The writer's tests in tension, however, show no effect of size of specimen whatsoever. Partly no doubt in consequence of this the suggested explanations for the size effect are utterly different: this is too complicated a subject for adequate discussion here, but each attempted explanation is available in the separate papers.

It might be not wholly unreasonable to add in relation to the author's model for the mechanical behavior of mild steel that his postulated two-component material is not wholly in conformity with modern dislocation theory and that it leads him into what appears to the writer to be a certain degree of illogicality in his conclusion that "the beginning of yielding may be expected to follow the same fundamental laws as the fracture of brittle materials." In fact, as the author knows and states, yielding can begin with equal ease in compression or tension. This of course is a reflection of the generally accepted fact that initial yielding depends on a function of the shear stresses and sc follows different fundamental laws from those which govern the fracture of brittle materials.

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[3] Professor of Mechanical Engineering, University Engineering Laboratories, University of Bristol, Bristol, England.

[4] G. Cook, "The Yield Point and Initial Stages of Plastic Strain in Mild Steel Subjected to Uniform and Nonuniform Stress Distributions," *Philosophical Transactions*, Royal Soc. (London), Vol. 230, p. 103.

[5] J. L. M. Morrison, "The Yield of Mild Steel, with Particular Reference to the Effect of Size of Specimen," *Proceedings*, Inst. Mechanical Engrs., Vol. 142, p. 193.

The writer would wish to thank the author for giving sufficient detail as to the conduct of his experiments (nowadays a very unusual, but still highly commendable, practice) to allow the reader to understand his methods and even perhaps to criticise them. Thus it seems to the writer that it would have been preferable to use specimens with enlarged ends since, even though similarity in relation to contact stresses was maintained, these contact stresses must surely weaken the beam and almost certainly to a different extent in cases where a drop in load is and is not found at yield. Again the author points out the variation in properties across a cross-section. Would it not have been better to machine all the small beams from the outer material which is most highly stressed in the larger beams? The author's statement on the difficulty of detecting the beginning of yielding is a fair and interesting one.

To the writer the most disappointing feature of the paper is the extremely large variation between the test results. By using statistical methods, the author claims to have eliminated the effect of all variables other than size and to find a positive effect of size whereby the largest beam tested yields at a stress about 70 per cent of that required to make his second-smallest beams yield. Taking, however, this second-smallest series of beams on its own, one finds that the weakest member yields at a stress of only 66 per cent of that required for the strongest, nominally identical, member. The writer does not for a moment suggest that the size effect is not valid. It would however, to him, have been more convincing had the control of other variables been more complete. The writer in fact finds it difficult to understand why a more complete control was not achieved. He would himself discard without hesitation as unsuitable for experimental purposes any material which gave uncontrollable variations of this order of magnitude.

The writer would very much regret it if the adversely critical tone of some of his comments were to be taken to imply that he felt on the whole adversely critical. The paper seems to him a valuable one and he looks forward with interest to seeing further test results which are promised in the conclusions.

MR. RICHARDS.—The author is aware of the publications of Cook in 1931 and Morrison in 1939. Both were referred to in the author's original work on the subject in 1953 (author's reference (**3**)). Little or no attention was paid to these papers, however, by those engaged in the controversy over the "raised yield point in bending." To the author's knowledge, he was the first to suggest any connection between the specimen size and the conflicting reports in this controversy. It was the possibility of such a connection that prompted the beginning of the author's studies in 1951.

The basic question of size effect itself could only be answered conclusively by tension tests, avoiding the complication of nonuniform stress distributions. A search of the literature showed that Mr. Morrison was the only one who even mentioned such tests for this purpose. It was soon found, however, that the evidence he presented was inconclusive. In the first place, the results of only three specimens are reported (Table 7 in Mr. Morrison's paper). Furthermore, his use of extensometers having knife edges probably affected the results. C. W. MacGregor[6] has shown that such knife edges materially lower the *upper* yield point, and the author discovered the same effect in his experiments. It was therefore decided to undertake a more thoroughgoing investigation of the possibility of size effect in simple tension. As a result of this research a definite size effect was discovered and reported in the author's previous paper in 1954 (author's reference (1)).

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[6] C. W. MacGregor, "The Yield Point of Mild Steel," *Transactions*, Am. Soc. Mechanical Engrs., Vol. 53, p. A-187 (1900).

The statistical theory, which offers an explanation for the phenomenon, thus was felt to be based on firm ground, *not depending on nonuniform stress distributions*. The present investigation had as its purpose an extension of the statistical theory to nonuniform stress.

The analogy of the beginning of yielding in mild steel with the fracture of a brittle material was more fully discussed in the author's previous paper (author's reference (1)). Briefly it is based on two points of similarity: both are triggered by the presence of flaws or imperfections in the material; and both are self-propagating, once started.[7] The analogy does not necessarily depend on the atomic mechanism involved, whether Cottrell atmospheres or brittle skeleton. Neither is it concerned with the type of stress responsible for failure.

The variation between the test results for a given size is primarily a consequence of the inherent variability in the upper yield point, which is due to the random distribution of imperfections as explained at the beginning of the paper. It should be remembered that the tests were designed to permit the material to attain the full value of the upper yield point, and the results were therefore subject to all of its inherent variations. In most commonly made tests no such precautions are taken and the upper yield point is masked to such an extent that it is the lower yield point that is really indicated. Since this is an entirely different phenomenon it is not subject to variations like those of the upper yield point. The noticeable deviations from linearity in the curves shown in Fig. 5 well below what might be called the yield stress, indicate the possibility that the full upper yield point was not reached. In the author's tests on beams in this size range the deviations from linearity were within 2 or 3 microinches per inch until abrupt yielding took place.

The author recognizes that, like any new theory, the statistical theory of the beginning of yielding in mild steel may be proven by later findings to be of limited applicability. The results so far, however, support the theory, and as Mr. Dolan points out, it has interesting possibilities. It may, for example, have some bearing on the notch brittleness of steel.

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[7] Cottrell, *Dislocations and Plastic Flow in Crystals*, Clarendon Press, Oxford, p. 145 (1953).

HYSTERESIS AND ANELASTICITY IN COLD-WORKED STAINLESS STEEL*

By J. D. Lubahn[1]

Synopsis

Creep and cyclic loading tests on cold-stretched stainless steel show that the hysteresis in cyclic loading is not an anelasticity phenomenon. The practical significance of this hysteresis as a possible source of damping is discussed.

The tests described in this report were carried out to learn the nature of the room-temperature hysteresis observed in cold-worked 12 per cent chromium stainless steel. Hysteresis is recognized experimentally by a loop in the stress-strain curve for constant loading and unloading rates (Fig. 1). Such behavior is of importance in many practical applications involving vibrations because a material with large hysteresis resists the buildup of serious vibration amplitudes under resonant or near-resonant conditions. When discussing these applications, the hysteresis is usually called "internal friction" or "damping."

The fact that cold working introduces hysteresis has been known for over 60 years (**1**)[2] and has been observed in many metals, but it is still poorly understood. It has been assumed, by Zener (**2**) for example, that this hysteresis is a manifestation of anelastic behavior, as are all hysteresis behaviors within the elastic limit. "Anelastic behavior" means that certain types of relationships will exist between stress, strain, and time. These relationships can all be derived (**2**) from the basic material behavior, expressed in terms of a "relaxation spectrum."

The relaxation spectrum is a graph used to describe the relative magnitudes of the coefficients in an infinite series of the form:

$$\epsilon/S = a_1(1 - e^{-t/\tau_1}) + a_2(1 - e^{-t/\tau_2}) + a_3(1 - e^{-t/\tau_3}) + \cdots + a_i(1 - e^{-t/\tau_i}) + \cdots \quad (1)$$

which is the formula for anelastic strain, ϵ, as a function of time, t, at a constant stress, S. In this equation, τ_i is called the "relaxation time constant" and is a measure of how slowly or quickly the anelastic strain appears. The coefficients a_i are a measure of how much anelastic strain (that is characterized by a certain time constant) will appear in infinite time (note that when $t = \infty$, each term reduces to a_i).

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* Presented at the Sixty-first Annual Meeting of the Society, June 22–27, 1958.

[1] Knolls Atomic Power Laboratory, General Electric Co., Schenectady, N. Y.

[2] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 980.

Since $(1 - e^{-t/\tau}) = 0.632$ when $t = \tau$, τ may be interpreted physically as the length of time needed for 63.2 per cent of the eventual anelastic strain to appear.

The behavior at constant stress can be expressed either as an infinite series or a finite series. When using an infinite series, each coefficient is an infinitesimal and the time constants in two adjacent terms differ by an infinitesimal. It has been shown (3) that the behavior can also be represented quite accurately by a finite series if the time constants in adjacent terms differ by a factor which is a small number, say, not over 10. The infinite series is used when expressing the behavior graphically as a relaxation spectrum. In this graph, the abscissa is the relaxation time on a logarithmic scale and the ordinate is arranged so that the area under the curve to the left of a certain time constant is the sum of the coefficients of all terms characterized by shorter relaxation times than the abscissa value. Depending on the source of the anelasticity (grain boundaries, thermoelastic effect, diffusion, etc.) the relaxation spectrum may be very broad, covering many log cycles of τ, as at elevated temperature (3); or it may have a quite sharp peak, so that the behavior can be described adequately by a single term of the above series, with a single relaxation time.

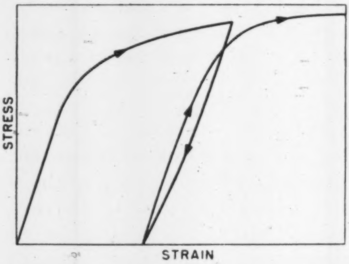

FIG. 1.—Schematic Diagram Showing Hysteresis Loop Caused by Cold Stretching.

Although the hysteresis introduced by cold work has always been assumed to be due to anelastic behavior, Zener (2) points out that "considerable uncertainty exists at present regarding the relaxation spectrum." From the fact (4–8) that the effects are independent of frequency, Zener has concluded that the relaxation spectrum is a horizontal line, but he hastens to cite one apparent exception (9) and points out that creep or relaxation experiments are needed to clarify the situation. The creep tests described below partially fulfill this need.

TESTS

The specimen was made from type 403 stainless steel quenched and tempered to 80,000 psi yield strength and stress relieved at 1175 F. The specimen was a standard 0.505-in. diameter, threaded-end bar provided with two SR-4 wire resistance strain gages placed on opposite sides of the bar. The loading sequence was:

1. Elastic loading to 30,000 psi (proportional limit = 65,000 psi approximately). Hold load at various levels and read gages. Average opposite gages.
2. Unload, using similar procedure.
3. Slow loading at constant rate to 83,000 psi (5500×10^{-6} plastic strain). Readings taken on the fly on the two gages alternatively. Simultaneous readings obtained by interpolation, and readings from opposite sides averaged.

4. Hold load constant at maximum value until creep rate becomes very small (approximately 1 hr).
5. Unload at a constant rate of 8000 psi per min.
6. Hold zero load until length remains constant.
7. Reload to 83,000 psi at a constant rate of 8000 psi per min to complete hysteresis loop.

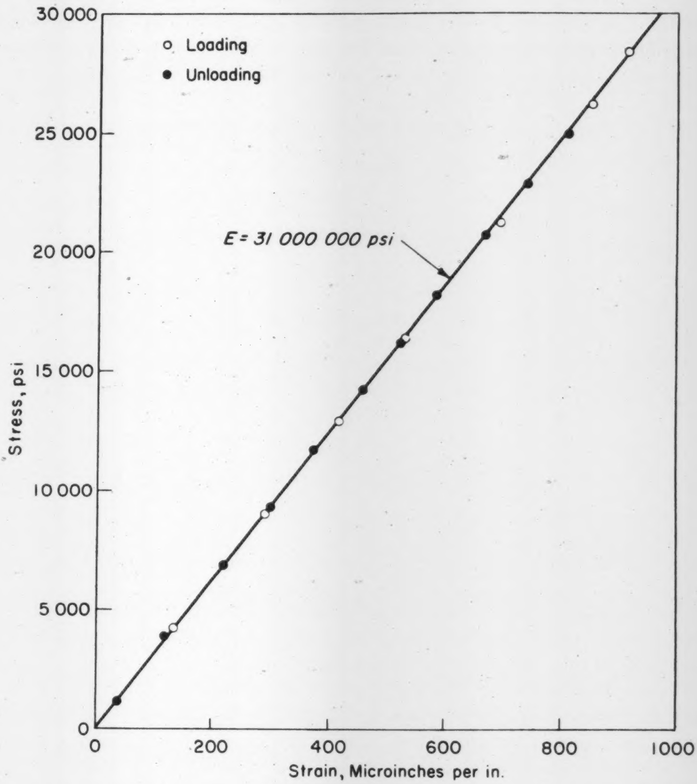

FIG. 2.—Elastic Loading and Unloading Curve for the As-Heat-Treated Material.

8. Hold maximum load until length remains constant.
9. Unload.
10. Hold 10 min and observe length changes.
11. Load suddenly to 55,700 psi and hold load constant 6 min.
12. Unload suddenly and observe subsequent length changes.

Figure 2 shows the elastic behavior of the virgin material. By the method used, no hysteresis could be detected. The elastic modulus was 31,000,000 psi.

Figure 3 shows the stress-strain curve into the plastic region. During the constant-rate loading there was 7100×10^{-6} plastic flow, as determined by the offset from the elastic line. There was an additional 1600×10^{-6} creep at maximum load, bringing the total plastic strain to 8700×10^{-6}. The unloading line was curved and less steep than the elastic loading line, and because of this, the plastic strain measured at zero load was only 8130×10^{-6}.

The details of the creep are shown in Fig. 4. The creep rate continuously diminished, but it had by no means ceased even after 1 hr.

Figure 5 shows the "elastic" unloading and loading curve after the plastic stretching shown in Figs. 3 and 4. The characteristic hysteresis loop was obtained. The initial portion of the loading curve had the same slope as the elastic curve for the virgin material. The same can be said of the unloading curve, but to a lesser degree of certainty. The total deviation from the elastic line amounted to 520×10^{-6}, or 20 per cent of the elastic strain. The width of the loop at the center is 190×10^{-6}.

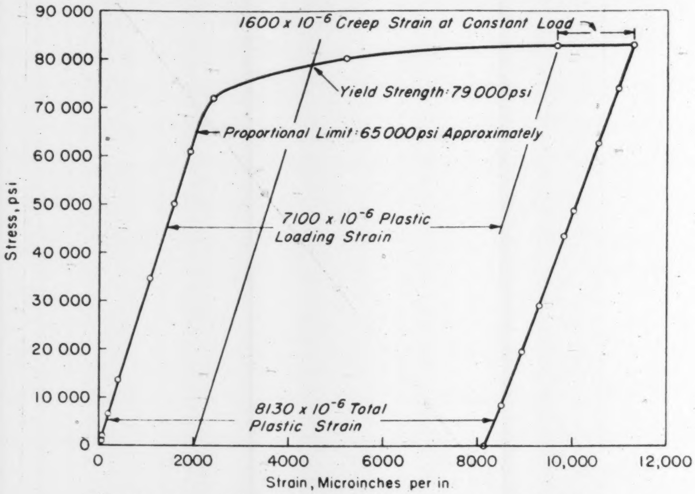

FIG. 3.—Stress-Strain Curve at Slow, Constant Strain Rate.

Note that the ends of the loop are very sharp, even though both minimum and maximum load were maintained for some time. In other words, at constant load there was no change in length that could be detected with a standard SR-4 indicator.

Figure 6 shows the results of suddenly loading to 56,000 psi and holding the load constant and of suddenly unloading and observing the subsequent length changes. The figure shows that there is no creep at constant load. Likewise, there is no change in length (called "creep recovery") at zero load. Of course, there may have been some very transitory effects, which had died out by the time the first reading was taken, but in that case the dying-out time was very short: less than 15 sec after loading and less than 6 sec after unloading.

From a cursory examination, the behaviors in Figs. 5 and 6 do not appear to be related to each other in terms of anelasticity. A pronounced hysteresis loop like that in Fig. 5 is generally regarded as being associated with an anelastic effect having a time constant of the order of the loading or unloading time (10 min). Figure 6, on the contrary, indicates that there is no anelastic strain, or at least none with a time constant longer than 10 sec or so.

ANALYSIS

Actually the test results of Figs. 5 and 6 are of such a nature that it is not convenient to compare them directly. Consequently an attempt was made to analyze and compare them in terms of a common quantity: relaxation time. In this analysis, it was assumed, for simplicity, that the constant-load behavior can be expressed in terms of a single time constant, τ:

$$\epsilon = aS(1 - e^{-t/\tau}) \qquad (2)$$

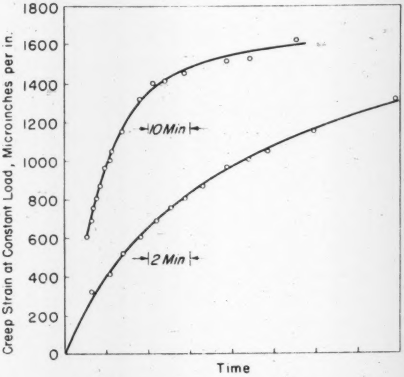

FIG. 4.—Creep Behavior Following Sudden Cessation of Loading at 83,000 psi in a Tensile Test (see also Fig. 3).

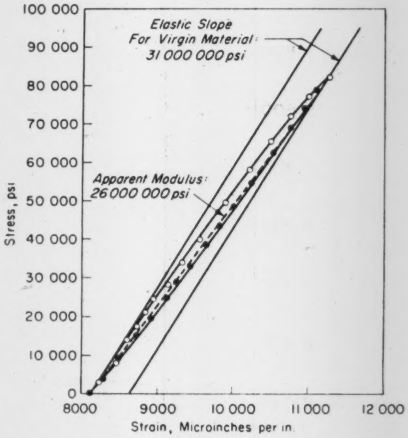

FIG. 5.—Hysteresis Loop in Stainless Steel After Plastic Stretching.

First we shall find the shape of the hysteresis loop for cyclic loading at constant loading and unloading rates $\dot{S} = dS/dt$, as shown in Fig. 7(*a*).

The strain, ϵ, at an arbitrary time, t, during the loading cycle AB due to the current loading cycle AB is:

$$\epsilon_1 = a\dot{S}[t - \tau(1 - e^{-t/\tau})] \qquad (3)$$

Reference 3 shows how this equation is calculated. Proceeding in a similar fashion, the strain, ϵ_2, at time, t, during the loading cycle AB due to all prior cycles ($n = 1$ to $n = \infty$) is:

$$\epsilon_2 = a\dot{S}\tau \left[\sum_{n=1}^{n=\infty} e^{-2n\Delta t/\tau}\right] [e^{-t/\tau}(e^{\Delta t/\tau} - 1)^2] \qquad (4)$$

Adding Eqs 3 and 4, the total strain at any time t during the loading cycle AB is:

$$\epsilon_{\text{loading}} = a\dot{S} \left\{[t - \tau(1 - e^{-t/\tau})] + \left[\sum_{n=1}^{n=\infty} e^{-(2n\Delta t)/(\tau)}\right] [\tau e^{-t/\tau}(e^{\Delta t/\tau} - 1)^2]\right\} \qquad (5)$$

The strain, ϵ_3, at an arbitrary time t during the unloading cycle BC due to the current unloading cycle BC is:

$$\epsilon_3 = -a\dot{S}[(t - \Delta t) - \tau(1 - e^{-(t-\Delta t)/(\tau)})] \quad \text{(6)}$$

The strain, ϵ_4, at time t during the unloading cycle BC due to the next preceding loading cycle AB is:

$$\epsilon^4 = a\dot{S}[\Delta t - \tau e^{-t/\tau}(e^{\Delta t/\tau} - 1)] \quad \text{(7)}$$

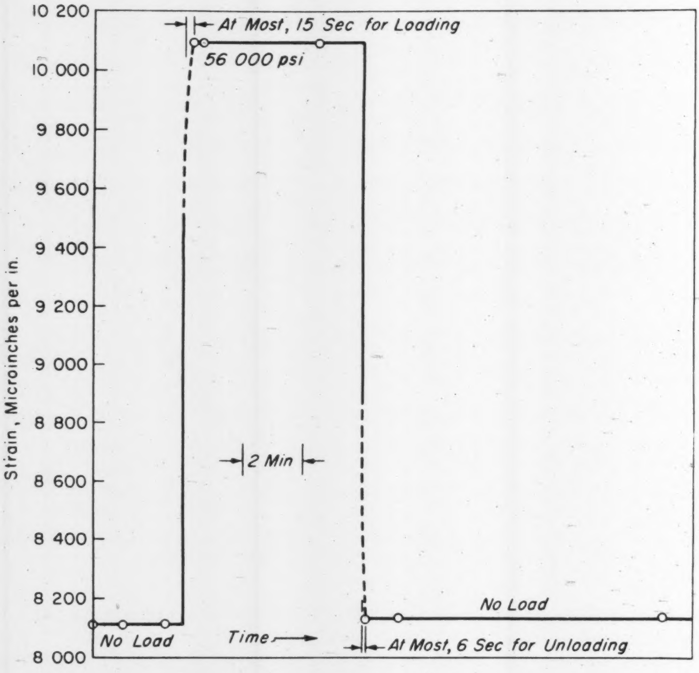

FIG. 6.—Creep Behavior at Constant Load Following Loading and Unloading.

The strain at time t during the unloading cycle BC due to all prior cycles ($n = 1$ to $n = \infty$) is given by Eq 4. Adding Eqs 4, 6 and 7 the total strain at arbitrary time, t, during the unloading cycle BC is

$$\epsilon_{\text{unloading}} = a\dot{S}\left\{[2\Delta t - t] + \tau[1 + e^{-t/\tau}(1 - 2e^{\Delta t/\tau})] + \left[\sum_{n=1}^{n=\infty} e^{-2n\Delta t/\tau}\right][\tau e^{-t/\tau}(e^{\Delta t/\tau} - 1)^2]\right\} \quad \text{(8)}$$

Equation 5 applies to values of t between zero and Δt, while Eq 8 applies to values of t between Δt and $2\Delta t$. These two equations are illustrated in Fig. 8 for $\tau = 10$ min and $\tau = 0.1$ min and for $\Delta t = 10$ min.

For the present purposes, we are concerned only with the shape of the hysteresis loop, that is, the width of the loop as a function of stress or time. The width δ is given by

$$\delta = (\epsilon_{\text{unloading}})_{t=t} - (\epsilon_{\text{loading}})_{t=2\Delta t - t} \quad \ldots\ldots (9)$$

$$\delta = a\dot{S}\tau \left\{ [2 + e^{-t/\tau}(1 - 2e^{\Delta t/\tau}) - e^{t/\tau}(e^{-(2\Delta t)/(\tau)})] + \left[\sum_{n=1}^{n=\infty} e^{-(2n\Delta t)/(\tau)} \right] [e^{-t/\tau}(e^{\Delta t/\tau} - 1)^2 - e^{t/\tau}(1 - e^{-\Delta t/\tau})] \right\} \quad \ldots (10)$$

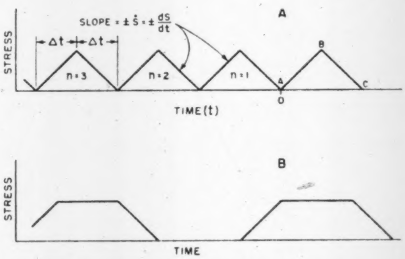

FIG. 7.—Two Load Histories Considered in the Analysis.

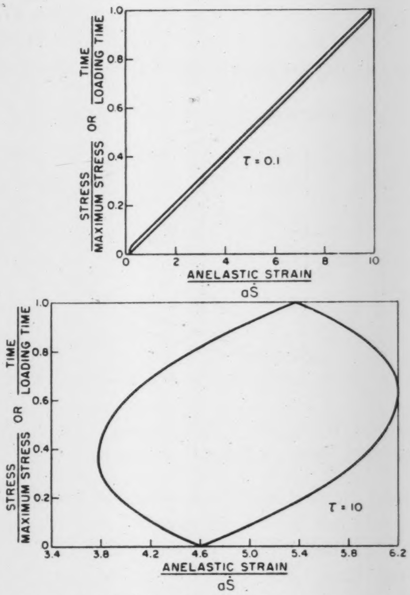

FIG. 8.—Calculated Hysteresis Loops (Anelastic Part) for Two Time Constants.

The width of the hysteresis loop as a function of stress or time is plotted in Fig. 9 for various values of τ, and the maximum width is shown in Fig. 10 as a function of τ. In both graphs $\Delta t = 10$ min as was the case in the tests. Note that the hysteresis loop has the characteristic lens-shaped appearance for values of τ greater than unity, but for smaller values of τ the sides of the loop are parallel except near the ends of the loop. Figure 8 shows an extreme example of the latter behavior for $\tau = 0.1$ min. From Fig. 9 it may be concluded that the hysteresis loop of Fig. 5 must correspond to a relaxation time of no less than 0.5 min, or 30 sec, if indeed it corresponds to any kind of anelastic behavior at all.

Now let us consider how small the relaxation time would have to be so that no creep and no creep recovery would be observed in a test like that shown in Fig. 6. Equation 2 shows the anelastic creep strain after time t at constant stress. The eventual anelastic creep (at $t = \infty$) is

$$\epsilon_{t=\infty} = aS \qquad (11)$$

Then the anelastic strain that will appear between $t = t$ and $t = \infty$ is the difference between Eqs 2 and 11, or:

$$\epsilon_{t=\infty} - \epsilon_{t=t} = aSe^{-t/\tau} \qquad (12)$$

To see this amount of creep or creep recovery, it would have to be larger than the limit of experimental accuracy of about 10×10^{-6}.

Equation 12 has been plotted in Fig. 11 for $t = 0.1$ (6 sec) and various values of τ. The value of a was obtained from Eq 10 and maximum loop width in Fig. 5. The value of 6 sec was chosen for t because there could have been anelastic strain occurring during an interval of as much as 6 sec between the beginning of unloading and the first reading at constant (zero) load. Figure 11 shows that the time constant could not have been over 0.7 sec, or creep recovery would have been observed after unloading. Similarly, considering the 15-sec loading period, the time constant could not have been over 1.8 sec, or creep would have been observed while holding the load constant at its maximum value.

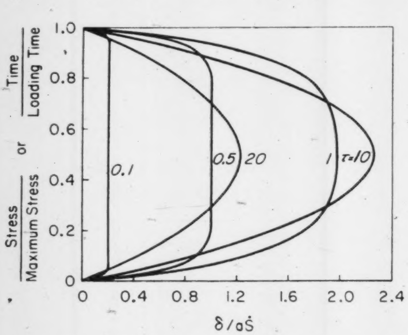

FIG. 9.—Width, δ, of Hysteresis Loop for Several Values of Time Constant, τ.

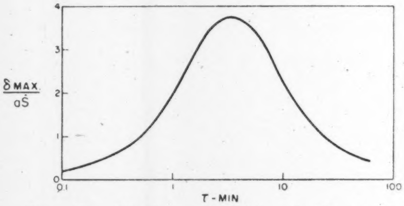

FIG. 10.—Maximum Width of Hysteresis Loop, $\delta_{\max}$ *versus* Time Constant, τ.

Summarizing, if the hysteresis in Fig. 5 were associated with anelasticity, the time constant would have to be greater than 30 sec, but the time constant would have to be less than about 1 sec to explain the behavior in Fig. 6. From these facts, it must be concluded that the observed hysteresis is not an anelasticity phenomenon. This conclusion is also consistent with observations that hysteresis in cold-worked metal is not a function of frequency.

Mention should also be made of two additional points of discrepancy between anelasticity and the observed behavior. First, anelasticity predicts a symmetrical hysteresis loop, while the observed loop is quite asymmetrical, being almost straight on the unloading side. Secondly, for the short time constants that are compatible with the behavior in Fig. 6, there is so much slope to the curve of anelastic strain *versus* stress that the apparent modulus (slope of the line through the ends of the loop) would be less than the initial modulus by much more than is actually observed. For $\tau = 0.1$ min, for example, Fig. 8 shows a tilt to the loop that spans 10 units of anelastic strain from minimum to maximum stress per 0.2 units of loop width, or a range of anelastic strain that is 50 times the loop width. For a measured loop width of 190×10^{-6} in Fig. 5, this corresponds to a range of anelastic strain of 50 by $190 \times 10^{-6} = 9500 \times 10^{-6}$. When added to the observed elastic strain (based on virgin material behavior) of 2500×10^{-6}, an apparent modulus only about one fifth the normal value would be obtained. On the other hand, the apparent modulus that is actually observed, Fig. 5, is only 16 per cent less than normal.

The calculated hysteresis loops shown in Fig. 9 are for continuous loading and unloading at constant rates, as shown in Fig. 7(*a*). The actual tests were performed with a period of constant load at zero and maximum, in the manner of Fig. 7(*b*). For these conditions the loading and unloading curves would be given by Eq 3, and there would be additional anelastic strain during the subsequent constant load period up to a total value of $aS\Delta t = 10a\dot{S}$, as shown in Fig. 12. Regardless of the value of τ, all these loops have flat horizontal ends comparable to the loop width. In other words, the anelastic strain at constant value of zero or maximum load is of the same order as the loop width. In contrast to this prediction from anelastic theory, the observed loop had very sharp ends, that is, no observable anelastic strain at constant value of zero or maximum load. From this comparison, it must also be concluded that the observed hysteresis is not an anelasticity phenomenon.

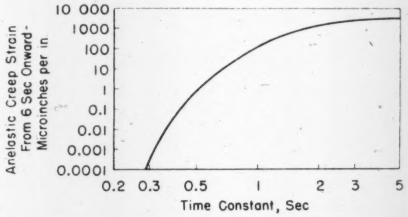

FIG. 11.—Effect of Time Constant on the Anelastic Strain at Constant Load Between t = 6 sec and $t = \infty$.

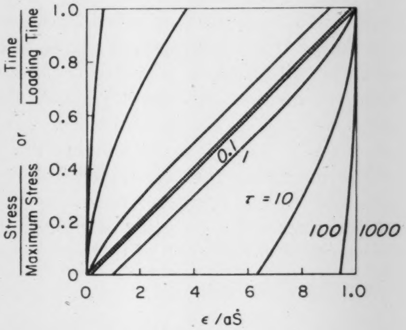

FIG. 12.—Shape of Hysteresis Loop (Anelastic Part) for Constant-Load Period at Maximum and Minimum Load.

SUMMARY AND DISCUSSION

All the characteristics of hysteresis in cold-worked metal are different from those expected from anelasticity theory:

1. The time constants for creep and cyclic loading are inconsistent with each other by at least a factor of 30.
2. The hysteresis loop is asymmetrical in shape.
3. The apparent modulus after cold work is much closer to the initial modulus than predicted by anelastic theory.
4. If the minimum and maximum loads are held constant, there should be anelastic strain, and this strain is not observed.
5. The hysteresis is not frequency dependent.

In the light of these discrepancies, it now appears necessary to reconsider the concept that hysteresis within the elastic limit is always an anelastic phenomenon. Much work will be required to bring to light the true nature of hysteresis caused by cold work.

Regarding the practical significance of hysteresis due to cold work as a source of damping in applications involving vibrations, but little can be said until the nature of the phenomenon has been explored more fully. One desirable feature is the insensitivity to frequency. The hysteresis caused by any anelasticity mechanism is at a maximum at a certain frequency and is much smaller at higher or lower frequencies. This means that the damping is only fully effective at a particular frequency, which may not be the operating frequency. The damping due to cold work, on the other hand, would be equally effective at any frequency.

The data on effects of temperature on hysteresis due to cold work are particularly confusing. From observations that a low-temperature heat treatment at zero stress removes the hysteresis observed during room-temperature vibration (10–11), it might be inferred that the hysteresis due to cold work would be of no value in applications involving vibrations at elevated temperatures, such as steam or gas turbine buckets. West (9), on the other hand, presents data to show that the hysteresis due to cold work *increases* as the temperature increases. Considerable experimentation will be necessary to clarify these temperature effects.

The magnitude of the hysteresis due to cold work can be compared with the hysteresis due to magnetostriction by means of the following analysis. The logarithmic decrement δ for magnetostrictive damping is about 0.03 (12). The logarithmic decrement δ corresponding to the hysteresis loop in Fig. 5 can be determined from the equation:

$$\delta = \frac{\text{energy dissipated in one cycle}}{2 \times \text{stored energy at one extreme of the cycle}}$$

or

$$\delta = \frac{\text{area in the loop}}{\frac{1}{2}\text{ area under the loop}}$$

Performing this operation with respect to Fig. 5 yields $\delta = 0.10$. Thus, the damping from cold work is three times the magnetostrictive damping.

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SUMMARY OF PROCEEDINGS OF THE SYMPOSIUM ON ADVANCES IN ELECTRON METALLOGRAPHY

This Symposium on Advances in Electron Metallography covers a broad range of electron metallography and is not concerned with electron microscopy alone. Four areas of investigation are included: (1) new improved techniques in specimen preparation and replication; (2) studies of the microstructure of age-hardenable and heat-resistant alloys; (3) electron probe microanalysis; and (4) direct transmission electron microscopy of metals. Electron metallography no longer includes only the areas of electron microscopy and diffraction but encompasses broadly all techniques which employ electron beam radiation to provide information on the nature of metals. This symposium publication, therefore, serves as a review of progress made in electron metallography during the past five years.

The symposium was developed under the sponsorship of Subcommittee XI on Electron Microstructure of Metals of ASTM Committee E-4 on Metallography. The papers in this symposium, together with the First Progress Report on the Non-Ferrous Task Group, were presented at a committee sponsored session Tuesday, June 24, 1958, during the Sixty-first Annual Meeting of the Society, Boston, Mass.

Mr. N. A. Nielsen, of E. I. du Pont de Nemours and Co., Inc., acted as Symposium Chairman, and Mr. Irwin Bessen, of North American Philips Co., Inc., presided over the session.

The papers presented were:

- Introduction—N. A. Nielsen
- Techniques Employed in the Electron Microscope Study of Titanium Alloy with 8 per cent Manganese—First Progress Report of Non-Ferrous Task Group of Subcommittee XI on Electron Microstructure of Metals
- Vibratory Polishing of Metallographic Specimens for Optical and Electron Microscopy—E. L. Long, Jr. and R. J. Gray
- Technique for Easy Removal of Direct Replicas for Electron Microscopy—W. H. Bridges and E. L. Long, Jr.
- Examination of Metals by Transmission Electron Microscopy—F. W. C. Boswell and E. Smith.
- A Study of Dislocations in Thin Aluminum Foils Elongated in the Electron Microscope—H. G. F. Wilsdorf
- Microstructure of Age-Hardenable Alloys—J. R. Mihalisin and K. G. Carroll
- An Electron Metallographic Study of the Precipitation-Hardening Process in Commercial Nickel-Base Alloys—W. C. Bigelow, J. A. Amy, C. L. Corey, and J. W. Freeman
- The Application of Electron Diffraction and Electron Microscopy in Studies of Minor Phases of Heat-Resistant Alloys—W. C. Bigelow, L. O. Brockway, and J. W. Freeman
- Electron Probe Analysis of Segregation in Inconel—L. S. Birks and E. J. Brooks

These papers have been issued as *ASTM Special Technical Publication No. 245* entitled "Symposium on Advances in Electron Metallography." The published volume includes a paper not presented at Boston: "Selected Etchants for Electron Microscope Studies of Magnesium Alloys," by C. A. Moe. The First Progress Report of the Non-Ferrous Task Group was published earlier in *Proceedings*, Am. Soc. Testing Mats., Vol. 57, p. 454 (1957).

SUMMARY OF PROCEEDINGS OF THE SYMPOSIUM ON ELEVATED TEMPERATURE STRAIN GAGES

The papers and panel discussions in this Symposium were presented at a special meeting sponsored by the Aeronautical Structures Laboratory, held at the Naval Air Material Center, Philadelphia, Pa., December 4–5, 1957. In view of the subject covered by this Symposium being of considerable concern to the ASTM-ASME Joint Committee on Effect of Temperature on the Properties of Metals, the Administrative Committee on Papers and Publications of ASTM sponsored its publication. D.E. Weiss acted as Technical Symposium Chairman and Commander H. J. Huester, Assistant Director of the Aeronautical Structures Laboratory, was general chairman.

The papers included in the Symposium are:

Introduction—D. E. Weiss
Recent Laboratory Evaluation of High-Temperature Strain Gages to 900 F—Emmett E. Day
Progress on Metalfilm Strain Gages—William T. Bean, Jr.
Advances in Static and Dynamic High Temperature Strain Gage Research—Richard H. Kemp
Temperature Compensation of High-Temperature Strain Gages—Edward I. Gray, Arthur Grossman, and Melvin R. Rubin
Laboratory Evaluation of Nichrome-Foil Strain Gage Installations up to 1200 F—William R. Forlifer
Optical Strain Gages for Use at Elevated Temperatures—H. K. Howerton
A Facility for the Evaluation of Resistance Strain Gages at Elevated Temperatures—R. L. Bloss
Resistance Measurement of Ceramic-Type Strain Gage Cements—J. W. Pitts, E. Buzzard, and D. G. Moore
Development of High-Temperature Strain Gage Wire—W. H. Graft
Evaluation of Several Gage Configurations Fabricated with Armour Alloy "D"—Jack J. Shrager
Ceramic Coatings for Experimental Stress Analysis—F. B. Stren, Jr.

Panel Discussion

Bonding Agents for High-Temperature Strain Gages—Dwight G. Moore
The Stability of Strain Gage Alloy Wires—Arthur G. Metcalfe
Foil Gages for High-Temperature Applications—Frank F. Hines
Temperature Compensation Techniques—Richard Friedman
Problems in Thermal Stress Measurements—D. J. DeMichele
Elevated Temperature Strain-Gage Investigations at Grumman Aircraft Engineering Corporation—Robert J. Stewart
High Temperature Strain Gage Evaluator—C. K. Martina
Bakelite Strain Gages for Temperatures Between −320 and 450 F—Donald J. Madsen
Investigation of Strain Gages for Long-Time Static Testing to 650 F—Herbert Yanowitz, Irwin Berman, and Alfred Bleiweis
High-Temperature Strain Gage Testing at Rolls-Royce—D. A. Drew

Additional Papers

Development and Application of High-Temperature Strain Gages for Stress Measurements in Jet Engines—B. R. Anderson
The Synthetic Mica Gage—G. A. Brewer

These papers with discussion were issued as *ASTM Special Technical Publication No. 230* entitled "Symposium on Elevated Temperature Strain Gages."

SUMMARY OF PROCEEDINGS OF THE SYMPOSIUM ON BRITTLE FAILURE OF ROTOR FORGINGS

The papers in this Symposium were presented at a Special Meeting at ASTM headquarters on November 11, 1957, for Delegates to World Metals Congress. It was sponsored by the Task Force on Brittle Fracture of Rotor Forgings of ASTM Committee A-1 on Steel.

The papers included in the Symposium are:

Inspection of Large Rotor Forgings—George E. Danner
Rotor Forgings for Power Equipment Manufacturers—T. G. Foulkes
The Work of the Task Group on Brittle Failure with Respect to Research—A. O. Schaefer
Requirements for Turbine and Generator Forgings—A. W. Rankin
Influence of Geometric Factors on Results of Ultrasonic Testing of Heavy Forgings—Helmut Krainer and Ekkehardt Krainer
The Axial Test Bore in Turbo-Generator Rotors —K. Kreitz

These papers were issued as *ASTM Special Technical Publication No. 231*, entitled "Symposium on Brittle Failure of Rotor Forgings."

SUMMARY OF PROCEEDINGS OF THE SYMPOSIUM ON SOLVENT EXTRACTION IN THE ANALYSIS OF METALS

Great interest has been engendered in the past several decades in solvent extraction separation methods. Their simplicity, speed, convenience, and versatility have earned for extraction techniques a favored place among separation methods of interest to analysts. The general aspects of metal extraction are discussed in this Symposium.

The papers, with discussions, were presented at the Fourth Session of the Sixty-first Annual Meeting of the American Society for Testing Materials, held in Boston, Mass., June 23, 1958, The Symposium was sponsored by ASTM Committee E-3 on Chemical Analysis of Metals through a Symposium Committee under the Chairmanship of H. Freiser, Department of Chemistry, University of Arizona. G. H. Morrison, Sylvania Electric Products, Inc., presided as Chairman of the Session.

The papers included in the Symposium were:

Introduction—H. Freiser and G. H. Morrison
Convergence of Tie Lines in Ternary Liquid Systems and Its Application to Liquid Extraction—R. L. Pilloton
Metals Analysis with Thenoyltrifluoracetone—F. L. Moore
The Use of Tri-*n*-Octylphosphine Oxide in Analytical Chemistry—J. C. White
8-Hydroxyquinaldine Extractions Applied to the Analysis of Metals—R. J. Hynek
Use of Organic Solvents in Flame Photometry—J. A. Dean

These papers with discussion were issued as *ASTM Special Technical Publication No. 238* entitled "Symposium on Solvent Extraction in the Analysis of Metals."

STUDIES OF CALCIUM SULFOALUMINATE ADMIXTURES FOR EXPANSIVE CEMENTS*

By A. Klein[1] and G. E. Troxell[2]

Synopsis

Various clinkers of theoretical calcium sulfoaluminate composition were produced and then studied by means of chemical analyses, differential thermal and X-ray analyses, and petrographic examinations to determine their compound composition. It was established that the raw materials did form a new compound not precisely identified, but which most likely was an anhydrous calcium sulfoaluminate.

A clinker burned at 2480 F, when ground and blended with slag and portland cement and used in 1:2.75 graded Ottawa sand mortars, cured 6 days in water and then dried in air 120 days developed final net expansions which ranged from 450 to 1800 millionths per unit. For a corresponding clinker burned at 2550 F, the final net expansions ranged from 5000 to 10,000 millionths per unit. In each case the coarser admixture showed the greatest net expansion. Somewhat smaller expansions in concrete may be sufficient for the "self stressing" of prestressed concrete.

The compressive strengths developed by some of the sulfoaluminate expansive cements were satisfactory for structural concrete.

The shrinkage of concrete has always been one of its greatest disadvantages for structures exposed to drying conditions. Investigations which have been made to determine the factors most responsible for the shrinkage of concrete have shown that, as affected by the cement, it is greater for fine-ground than for coarse portland cements, for type IV (low-heat) than for type I (normal) cement, for high water-cement ratios and for high cement contents. Shrinkage is also markedly affected by the mineralogical character of the aggregate.

Shrinkage is caused primarily by the loss of moisture under drying conditions, but the concrete will swell again when it reabsorbs water. Hence the volume changes are reversible, at least approximately, depending upon the ambient moisture conditions.

European Investigations

During the past twenty years, investigators in Europe, notably Lossier, Lafuma, Perre, and Mikhailov, have directed their attention to the possibility of eliminating shrinkage, or even causing the concrete to show an appreciable net increase in length, by using sulfoaluminate admixtures in cements to cause controlled expansions.

The earliest publication on sulfoaluminate admixtures is the French patent issued in February 1936 to Etablissements Poliet & Chausson, Paris. The most aggressive investigator of the admixtures covered in the patent has been Henry Lossier (1),[3] of France, and expansive cements containing these admixtures are known in Europe as Lossier cements.

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According to this process, the use of sulfoaluminate admixtures in the amount of 10 to 20 per cent by weight of portland cement results in a controllable expansion to overcome or to exceed the shrinkage produced by the consolidation of the colloidal gels during the setting and hardening processes and their subsequent drying. These sulfoaluminates, whether produced by a fusion process or by mixing of calcium sulfates and aluminates, are presumed to react with the aluminates already present in the portland cement to produce hydrated sulfoaluminates with a large number of water molecules. It is indicated in the literature that to balance total portland cement shrinkage at a given age a sulfoaluminate rich in SO_3 and sparse in CaO is desirable, forming the low sulfate $3CaO \cdot Al_2O_3 \cdot CaSO_4 \cdot 12H_2O$. Where it is desired to have high expansions to develop prestress, or so-called "self-stress" in steel, it is desired to use a composition such as to produce the more highly expansive form of sulfoaluminate—the high sulfate, $3CaO \cdot Al_2O_3 \cdot 3CaSO_4 \cdot 30$ to $32H_2O$.

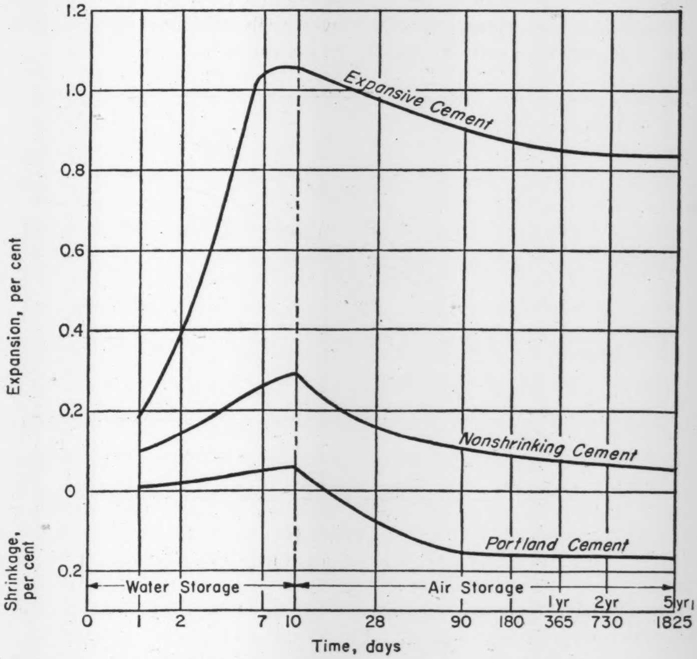

FIG. 1.—Volume Changes of Neat Cement Pastes (1).

Recently, V. V. Mikhailov (2), of the U.S.S.R., in presenting new developments in self-stressed concrete, indicated the advantages of a prehydration procedure for a portion of the sulfoaluminate admixture in a manner to allow control of the reactions between the hydrates of the high and low sulfates and to delay the formation of the highly expansive high sulfate until the concrete had developed considerable strength. The Russian expansive cement is produced by the dry intergrinding of portland cement, aluminoferrous cement, and gypsum, within limits. Figure 1 shows the general effect upon volume changes of neat cement pastes when using these materials. For a 1:6 concrete mix, the expansion would be only about one third that for a cement paste. Figure 2 shows the corresponding strengths of 1:3 plastic mortars (1). In comparison with portland cement, expansive cements develop strength a little more slowly at first, but eventually their strength surpasses that of the portland cement.

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[3] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 1008.

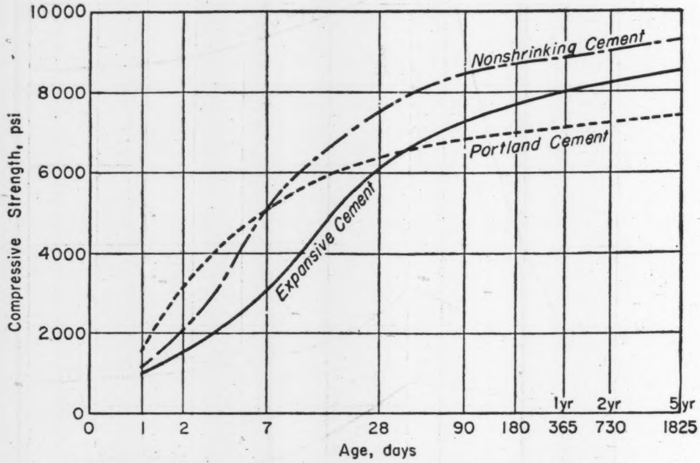

FIG. 2.—Compressive Strength of 1:3 Plastic Mortar Cubes (1).

whereas the Lossier cements are produced by the intergrinding of portland cement, a fusion product claimed to be a sulfoaluminate composition, and a blast-furnace slag.

Characteristics of European Expansive Cements:

It has been found that concretes made with the European expansive cements shrink upon drying, just like conventional concrete, but by undergoing an initial expansion of a controlled amount before drying begins, the net expansion after drying may be any desired amount

It was noted that if the expansion occurred too rapidly, the bond between the particles was not strong enough to hold them together, so disruptions of the mass occurred. By adequate curing to develop strength to resist disruption and by control of the rate and amount of expansion, a sound expanded product was obtained.

Some investigators have suggested that with larger amounts of the sulfoaluminate component, it would be possible for the expansion of the concrete to develop tensile stresses in embedded steel and thus automatically to produce the equivalent of prestressed concrete. Although the longitudinal restraint of the steel strands causes some longitudinal compression to develop in the concrete, a large part of this compression may later be lost due to shrinkage and creep in the concrete, as this compression is developed at an early age.

One of the advantages of expansive cements is said to be the superior quality of the resulting concrete with respect to permeability. This results from the closure of some voids by the expansion of the cement and from the elimination of incipient contraction cracks during the aging of the concrete (3).

One disadvantage is that expansive cements deteriorate rapidly unless properly protected from humid air. For that reason they should be packaged in moisture-proof bags. Another disadvantage is that they are not resistant to sea water or to water containing sulfates. Furthermore, aggregates used with expansive cements should contain no soluble sulfates (1).

Calcium Sulfoaluminates:

It is known that the calcium sulfoaluminates have a major effect in the setting process of portland-cement pastes and constitute a considerable portion of the material hydrated up to the age of about 12 hr. Eitel (5), Jones (6), Kalousek (7) and others have investigated the system lime-alumina-calcium sulfate-water with respect to the formation of the so-called high-sulfate ($C_3A \cdot 3CaSO_4 \cdot 32H_2O$) and low-sulfate ($C_3A \cdot CaSO_4 \cdot 12H_2O$) hydrates of the calcium sulfoaluminates. However, very little information is available with respect to the structure of these hydrates as defined by differential thermal analyses, X-ray diffraction spectra, and optical constants.

It appears that the sulfoaluminate clinker utilized in the manufacture of Lossier expansive cement should provide a source of the anhydrous compound for studies of its properties, the manner of hydration, and its influence on the hydration characteristics of portland cement. An examination of this anhydrite has been conducted by Lafuma (3). His investigations of clinkers of commercial production, employing X-ray analysis, indicated that, instead of calcium sulfoaluminate, the composition consisted of about equal parts of free anhydrous calcium sulfate and calcium aluminate—particularly C_5A_3—in the amount of about 75 per cent of the total clinker. He concluded that the same effects could be obtained in the production of hydrates, such as the high sulfate ettringite ($3CaO \cdot Al_2O_3 \cdot 3CaSO_4 \cdot 32H_2O$) through the use of a mixture of high-alumina cement and calcium sulfate, or by burning a properly proportioned mixture of gypsum and bauxite.

The composition cited above, calculated to a basis free of impurities is 50 per cent CaO, 24 per cent Al_2O_3, and 27 per cent SO_3. Lafuma states that this composition is rather high in lime from the point of view of a mixture containing anhydrous calcium sulfate and calcium aluminates. However, from the point of view of a pure tetracalcium sulfoaluminate, which would contain 55 per cent CaO, 25 per cent Al_2O_3, and 20 per cent SO_3, it is calculated that the Lossier admixture contains a small excess of Al_2O_3 (1 per cent) and a large excess of SO_3 (9 per cent).

In the utilization of sulfoaluminate clinkers in expansive cements, there is employed a so-called "terminator" or "stabilizer," such as a blast-furnace slag, in addition to the major portland cement constituent. It is said that the mechanism of termination of the expansive reaction is the absorption of excess calcium sulfate by the slag (3). Consequently, for a given portland cement, the amount, fineness, and the nature of the terminator employed will be of importance, as well as the amount of free lime present both in the portland cement and in the sulfoaluminate clinker. According to M. E. Perre (4), "Sulfoaluminate cement is extremely sensitive to the presence of free lime, a small quantity of which is sufficient to cause disintegration."

TABLE I.—RAW-MIX PROPORTIONS AND COMPOSITIONS FOR POTENTIAL CALCIUM SULFOALUMINATE CLINKERS.

	Clinker A	Clinker B	Clinker C	Clinker D	Clinker E	Clinker F
Raw mix proportions, parts by weight						
Raw basis:						
Reagent quality						
Gypsum	0.355	0.076	...	...	...	...
Alumina	0.225	...	...	...	...	...
Calcium hydroxide	0.420	0.647	...	...	...	...
Aluminum sulfate (anhydrite)	...	0.277	...	...	...	...
Calcite	...	...	0.255	0.558	0.421	0.406
Industrial gypsum	...	...	0.376	...	...	0.188
Industrial aluminum sulfate (hydrate)	...	...	...	0.280	0.366	0.140
South American bauxite	...	...	0.369	0.162	0.213	0.266
Ignited basis:						
Calcite	...	...	0.211	0.536	0.400	0.373
Industrial gypsum	...	...	0.427	...	...	0.214
Industrial aluminum sulfate (hydrate)	...	...	...	0.279	0.360	0.140
South American bauxite	...	...	0.362	0.185	0.240	0.273
Computed potential oxide analysis, percentage by weight						
SiO_2	...	...	3.2	1.6	1.8	2.4
Al_2O_3[a]	25.1	25.1	31.1	24.6	31.7	27.8
Fe_2O_3	...	...	1.4	0.9	1.2	1.1
CaO	55.2	55.2	37.9	51.7	38.6	44.8
MgO	...	...	0.3	0.9	0.7	0.6
SO_3	19.7	19.7	24.4	19.2	24.8	21.8
Undetermined	...	...	0.1	0.3	0.1	0.3
Potential compound composition, excesses above composition $4\,CaO \cdot Al_2O_3 \cdot SO_3$, per cent						
Al_2O_3	None	None	13.8	1.1	14.2	7.5
SO_3	None	None	10.8	0.7	11.0	5.8
Temperature for incipient fusion of raw mix						
deg Fahr	...	...	2610	2550	2600	2490
deg Cent	...	...	1430	1400	1430	1370

[a] Includes TiO_2.

In summarizing the literature with respect to the composition of sulfoaluminate clinkers it appears that, according to Lafuma, the clinker is principally a mixture of anhydrous calcium sulfate and the calcium aluminate C_5A_3. This would not rule out the possible presence of a significant amount of a calcium sulfoaluminate as high-temperature equilibria have not been determined for the ternary system $CaO\text{-}Al_2O_3\text{-}SO_3$, and X-ray diffraction patterns for any ternary compounds are not available in the literature.

CURRENT RESEARCH PROGRAM

As the available references on the European investigations do not give any information concerning solid-state thermal reactions for the ternary system $CaO-Al_2O_3-SO_3$, or other essential details on the manufacture of expansive cements, the present study was undertaken to produce various sulfoaluminate admixtures and to determine their characteristics in combination with portland cement.

The investigation involved the production of a preliminary sulfoaluminate composition, using pure materials in two different combinations which were burned in a differential thermal analysis (DTA) furnace, and the production of four additional compositions made from commercial materials, small amounts of which were burned in an electric furnace and larger amounts of which were burned in a rotary kiln.

Differential thermal analyses were made on each raw mix to determine any critical temperature ranges which could indicate the formation of new compounds. Also, X-ray diffraction patterns were determined for each composition burned at each critical temperature to determine what new compounds, if any, were formed, and their temperature of formation.

Each material burned in the kiln was ground to three degrees of fineness. Each ground material was then blended with ground blast-furnace slag and used to replace various percentages of either of two brands of portland cement of markedly different compositions, one of low C_3A and low alkali content and the other of high C_3A and high alkali content. The resulting cements were tested by noting the expansive characteristics and, for some conditions, the compressive strength of mortars in which they were used.

Manufacture of Sulfoaluminate Clinker from Reagent-Quality Chemicals:

In order to investigate the products of thermal reactions at temperatures up to 1300 C, raw mixes were made with reagent quality materials proportioned to yield, on the ignited basis, an oxide composition equivalent to that of the compound $4CaO \cdot Al_2O_3 \cdot SO_3$. Two raw-mix compositions were designed, employing different raw-mix ingredients, to yield the same oxide composition upon firing. The first raw-mix composition, designated as composition A in Table I, utilized reagent quality $CaSO_4 \cdot 2H_2O$ (gypsum), Al_2O_3 (alumina), and $Ca(OH)_2$ (hydrated lime) in theoretical proportions to yield a fired composition of 55.2 per cent CaO, 25.1 per cent Al_2O_3, and 19.7 per cent SO_3. The second raw-mix composition B utilized reagent quality gypsum, hydrated lime, and anhydrous aluminum sulfate in the required theoretical proportions to yield the same composition on firing as that for composition A.

A differential thermal analysis was made for each raw mix composition to record its complete thermal history to about 1340 C. Then, by heating successive separate samples of each composition to a temperature just beyond each reaction peak, and making an X-ray analysis of each product, it was possible to determine the cause of most of the peaks. Similar differential thermal analyses were made for each of the raw-mix ingredients—gypsum, hydrated lime, and aluminum sulfate. The thermograms for the raw materials and the two raw-mix compositions are shown in Fig. 3.

The pronounced endothermic peak at about 560 to 570 C for each of the raw-mix compositions noted in Fig. 3 was caused by the decomposition of the hydrated lime, as indicated in the thermogram for calcium hydroxide. The thermograms for both compositions A and B show an endothermic reaction between 1210 and 1240 C attributed to inversion of calcium sulfate from the monoclinic to the rhombohedral form.

No changes were observed in the X-ray patterns for either composition in samples heated to 700 C and to 980 C which would account for the endothermic reaction peaks at about 840 C. The X-ray diffraction patterns for each composition heated above 1300 C disclosed interplanar spacings for a compound not listed in published literature on X-ray diffraction data. The unknown compound was a major constituent, and the remainder appeared to consist of a considerable amount of rhombohedral calcium sulfate, and some free CaO. The X-ray diffraction spectra for the unknown compound in both compositions were, in decreasing order of intensity:

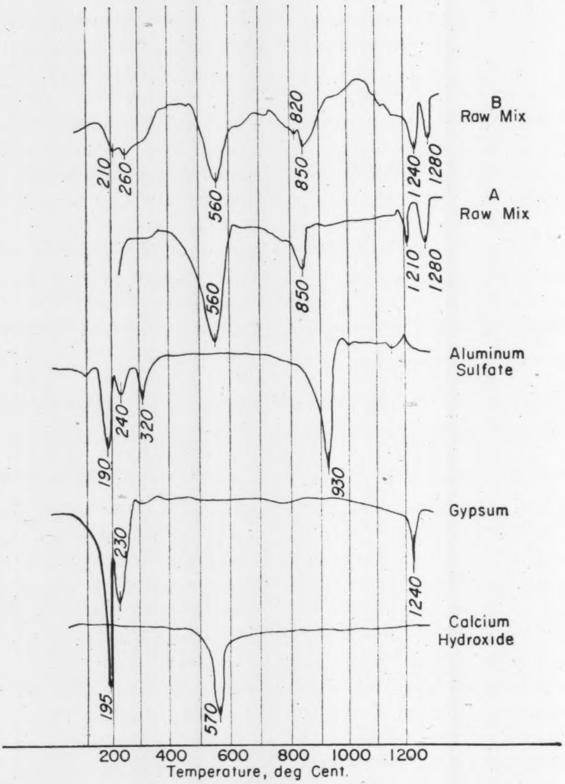

FIG. 3.—Differential Thermal Analysis Curves for Raw Materials and Raw Mixes A and B.

d, Å....	3.75	3.24	2.90	2.65	2.16

The reaction peaks on the thermograms at 840 C may indicate the formation of nuclei of the compound but in such small amounts that it was not detected by X-ray. It was observed that the spacings noted above could be detected for samples heated to 1050 C in the DTA furnace. Not all the ingredients had reacted even at 1350 C, indicating that more hold time is required for complete reaction.

While in this case, quantitative estimation of composition from X-ray patterns is unreliable, it is roughly estimated from the X-ray patterns that each of the compositions, regardless of starting materials, contained in addition to about 5 per cent free CaO, about equal parts of an unknown compound and calcium sulfate. The relative proportions of the reaction products obviously would be different under conditions of longer soaking at the temperature of incipient fusion.

TABLE II.—CHEMICAL ANALYSES OF MATERIALS USED IN INVESTIGATION.
Percentage by weight.

	Portland Cement		Commercial Lossier Cement	Raw Materials for Manufacture of Sulfoaluminate Clinkers[b]			
	No. 1	No. 2		Calcite	Gypsum	Bauxite	Aluminum Sulfate
SiO_2	24.0	21.3	19.0	0.8	2.0	6.0	None
Al_2O_3[a]	2.7	6.4	8.8	0.4	0.1	85.4	30.6
Fe_2O_3	2.8	2.7	2.6	0.0	0.1	3.5	0.8
CaO	66.8	62.6	59.5	97.5	41.0	0.4	None
MgO	0.7	2.7	1.7	1.2	0.0	0.0	None
SO_3	1.7	2.2	5.7	...	56.9	...	...
Na_2O	0.1	0.6	0.1	...	...	...	...
K_2O	0.1	0.8	0.3	...	...	...	...
Loss on ignition	0.9	0.9	1.9	43.6[c]	20.7[c]	31.7[c]	40.2[c]
Free CaO	0.5	0.2	2.1	...	...	...	...
Acid insoluble in 1:3 HCl	...	...	4.2	...	...	...	...
C_3S	60	39					
C_2S	22	31					
C_3A	2	12					
C_4AF	8	8					

[a] Includes TiO_2.
[b] Analyses calculated to fired basis.
[c] Loss on firing to 2000 F.

Manufacture of Sulfoaluminate Clinker from Materials of Industrial Quality:

Based upon the results obtained in the manufacture of compositions A and B, further tests were made employing raw materials of industrial quality. The methods of manufacture included the preliminary use of a DTA furnace, of an electric furnace heated by globar elements, and of a rotary kiln.

Four raw-mix compositions were designed, employing different combinations of raw materials, to produce clinkers having nominal CaO contents of 38, 45, and 52 per cent. As shown in Table I, the raw mix for composition C contained industrial calcite, gypsum, and South American bauxite while the raw mix for composition E contained calcite, aluminum sulfate, and bauxite. These compositions were designed for a potential oxide composition of about 38 per cent CaO, 31 per cent Al_2O_3, and 25 per cent SO_3. It is noted that such a potential oxide composition provides Al_2O_3 in excess of about 14 per cent, and SO_3 in excess of about 11 per cent, as compared to the requirements for the compound

$4CaO \cdot Al_2O_3 \cdot SO_3$ (also expressed as $C_3A \cdot CaSO_4$).

The raw mix for composition D, as for composition E, contained industrial calcite, aluminum sulfate, and bauxite, but was proportioned to provide a potential oxide composition of about 52 per cent CaO, 25 per cent Al_2O_3, and 19 per cent SO_3. For this potential composition, Al_2O_3 and SO_3 were present in excess of only about 1 per cent. The raw mix for composition F contained calcite, gypsum, aluminum sulfate, and bauxite, and was proportioned to provide a potential oxide composition of about 45 per cent CaO, 28 per cent Al_2O_3, and 22 per cent SO_3. In this mix Al_2O_3 would be in excess by about 8 per cent and SO_3 by 6 per cent with respect to the composition of the pure compound $C_3A \cdot CaSO_4$.

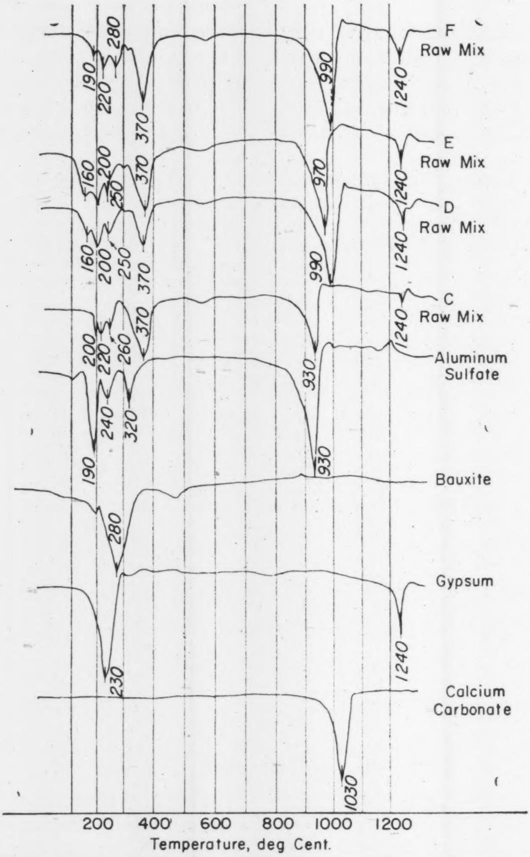

FIG. 4.—Differential Thermal Analysis Curves for Raw Materials and Raw Mixes C, D, E and F.

The chemical analyses of the raw materials are shown in Table II, together with a chemical analysis of Lossier expansive cement and of two portland cements included in subsequent research covered in this report.

Burns in Differential Thermal Analysis Furnace.—The differential thermal analysis curves for the raw materials employed in producing raw mixes for compositions C, D, E, and F are shown in Fig. 4. Also shown are the curves obtained for burning each of the raw-mix compositions in the DTA furnace. The endothermic reactions obtained below 400 C upon burning the several raw-mix compositions corresponded in general to reactions taking place in the raw-material components due to losses of water.

TABLE III.—OXIDE COMPOSITION OF HARD-BURNED CALCIUM SULFOALUMINATE CLINKERS MANUFACTURED IN GLOBAR ELECTRIC FURNACE.

Percentages by weight.

Oxide Analysis, Ignited Basis	Clinker C	Clinker D	Clinker E	Clinker F
SiO_2	3.4	1.7	2.0	2.0
Al_2O_3[a]	36.3	27.4	37.0	32.6
Fe_2O_3	1.6	0.8	1.2	1.1
CaO	41.1	51.6	39.7	45.1
MgO	0.3	0.6	0.5	0.5
SO_3	16.6	17.8	19.8	18.3
Na_2O	0.0	0.0	0.0	0.0
K_2O	0.1	0.0	0.0	0.0
Total	99.4	99.9	100.2	99.6
Loss on ignition	0.3	0.7	0.5	0.4
Acid insoluble in 1:3 HCl	0.6	0.3	0.7	0.8
Free CaO	0.1	15.9	0.0	2.6

[a] Includes any TiO_2.

The raw-mix compositions C, D, E, and F differed significantly from those for compositions A and B in that calcium carbonate was employed instead of calcium hydroxide. Hence, calcium oxide became available for thermal reactions only at a substantially higher temperature than in the burning of compositions A and B. This factor is reflected in the thermograms in that the maximum endothermic reactions for compositions A and B occurred at temperatures from 100 to 150 C lower than for raw mixes containing calcium carbonate. It is believed that the endothermic reactions between 930 and 990 C corresponded to the start of loss of carbon dioxide from the calcite constituent together with considerable nucleation of the ultimate calcium sulfoaluminate component of the clinker. It is noted also that while the inversion of calcium sulfate was again indicated at 1240 C, the magnitude of the reaction was quite small compared to that between 930 and 990 C, whereas for raw-mix compositions A and B the magnitude of the inversion reaction appeared to be the major reaction. This indicated that the proportion of $CaSO_4$ in the clinker was quite low for compositions C and D, and somewhat higher for compositions E and F.

Burns in Electric Furnace.—The raw mixes for compositions C, D, E, and F were molded with the aid of water into bars $\frac{1}{2}$ in. square and 4 in. long. After oven drying, the bars were placed in the form of a grillage on a periclase hearth brick, and each composition was burned separately in an electric furnace at a maximum temperature of 1350 C (2460 F). The temperature in each case was raised at a rate such as to obtain the maximum temperature at 1 hr after start of heating, and the bars were soaked at the maximum temperature for 20 min. The temperature was dropped to 1000 C over a period of 10 min, and the hearth supporting the bars was thereafter removed for rapid air-quenching.

It was observed that loss of sulfur dioxide commenced upon heating to about 1000 C, with a maximum rate of evolution at about 1340 C. A chemical analysis shown in Table III was made for each of the clinkers produced.

Burns in Rotary Kiln.—A series of burns, each lasting about 12 hr, was performed in a rotary kiln which was of 30-ft length and 21-in. inside diameter, with a $4\frac{1}{2}$-in. periclase brick lining in the 10-ft burning zone. The kiln was operated at a speed of 1 rpm and on a slope of 0.5 in. per ft. All burning was conducted under oxidizing conditions at a draft of 0.06 in. of water maintained by automatic draft control. The kiln feed was prepared by nodulizing the raw mix to provide particles between $\frac{1}{4}$-in. and $\frac{1}{2}$-in. diameter with a water content of about 15 per cent. Raw nodules were fed to the kiln at a rate of about 200 lb per hr. Discharged clinker was rapidly cooled by means of an air blast. The time of residence for material being burned was about 1 hr and 15 min. For each of the four compositions C, D, E, and F, about 800 lb of clinker was produced at the temperature of incipient fusion. For compositions C and D, a smaller amount of material was produced under a condition of soft burning by dropping the temperature about 50 C below that of incipient fusion.

Chemical analyses were made for each of the six clinkers produced by burning in the rotary kiln. A comparison of the actual oxide compositions from Table IV with the potential oxide compositions from Table I is shown in Table V. It is clear that harder burning resulted in greater loss of sulfur dioxide and reduction from the potential SO_3. It is noted also that the oxide analysis for composition D, soft burned in the rotary kiln at 2480 F (Table IV), is almost identical to the oxide analysis of the same composition burned in the electric furnace at 2460 F (Table III).

It appears that harder burning results in greater amounts of free CaO, possibly due to dissociation of some $CaSO_4$ present in the clinker, or due to excess lime in the composition with respect to the stoichiometry of the actual calcium sulfoaluminate formed. This condition is also shown for composition F, where the rotary-kiln product contained 5.1 per cent free CaO as compared to 2.6 per cent free CaO for the same composition burned at lower temperature in the electric furnace. It is noted also that the higher the potential CaO content for a given composition, the higher the actual free CaO in the clinker. It appears, therefore, that raw mixes high in lime are

TABLE IV.—OXIDE COMPOSITION OF CALCIUM SULFOALUMINATE CLINKERS MANUFACTURED IN ROTARY KILN.

Percentages by weight.

Oxide Composition	Clinker C		Clinker D		Clinker E	Clinker F
	Hard Burned	Soft Burned	Hard Burned	Soft Burned	Hard Burned	Hard Burned
SiO_2	3.7	3.4	1.4	2.3	2.2	2.8
Al_2O_3[a]	37.8	36.5	26.4	25.0	37.4	29.3
Fe_2O_3	1.5	1.4	1.0	2.0	1.4	1.4
CaO	42.9	40.6	54.3	51.6	44.4	44.4
MgO	0.6	0.5	0.9	0.6	0.7	0.7
SO_3	13.6	17.2	15.9	17.7	13.9	21.2
Loss on ignition	0.2	0.2	0.4	0.4	0.5	0.3
$Na_2O + K_2O$	0.0	0.0	0.0	0.0	0.0	0.0
Total	100.3	99.8	100.3	99.6	100.5	100.1
Insoluble in 1:3 HCl	0.6	0.8	0.2	0.6	0.3	0.8
Free CaO	0.1	0.1	20.8	16.7	0.0	5.1
Temperature of burning, deg Fahr	2610	2530	2550	2480	2620	2500

[a] Includes any TiO_2.

most likely to produce clinker containing principally calcium sulfoaluminate and free lime, with a minimum of calcium sulfate. It is also quite possible that the high free lime contents associated with the high CaO contents are indicative of a ternary compound of different stoichiometry than the $C_3A \cdot CaSO_4$ compound assumed in the design of the raw mixes.

TABLE V.—POTENTIAL AND ACTUAL OXIDE COMPOSITIONS OF CLINKERS PRODUCED IN ROTARY KILN.

Oxide		Clinker C		Clinker D		Clinker E	Clinker F
		Hard Burned	Soft Burned	Hard Burned	Soft Burned	Hard Burned	Hard Burned
CaO	Potential	37.9	37.9	51.7	51.7	38.6	44.8
	Actual	42.9	40.6	54.3	51.6	44.4	44.4
Al_2O_3	Potential	31.1	31.1	24.6	24.6	31.7	27.8
	Actual	37.8	36.5	26.4	25.0	37.4	29.3
SO_3	Potential	24.4	24.4	19.2	19.2	24.8	21.8
	Actual	13.6	17.2	15.9	17.7	13.9	21.2
Totals	Potential	93.4	93.4	95.5	95.5	95.1	94.4
	Actual	94.3	94.3	96.6	94.3	95.7	94.9
Free CaO	Actual	0.1	0.1	20.8	16.7	0.0	5.1

Composition D most nearly approached the composition (calculated free of impurities) for the expansive admixture of Lossier cement described by Lafuma. The table below illustrates the similarity in compositions.

Material	CaO	Al_2O_3	SO_3	Free CaO
Lossier admixture, calculated free of impurities	50	24	26	[a]
Composition D				
Hard burn	54	26	16	21
Soft burn	52	25	18	17

[a] Claimed to be low.

It appears that the expansive admixture for Lossier cement was considerably underburned, and it is logical to expect that the excess SO_3 was present as $CaSO_4$. It is believed that successful commercial operations to produce material of uniform quality must be based on the burning of all clinker to incipient fusion as is done for portland cement.

X-ray Analysis of Sulfoaluminate Clinkers:

Analysis by X-ray diffraction was made for all of the clinkers produced by burning in the DTA furnace, the electric furnace, and in the rotary kiln. The X-ray patterns represented only the front-reflection region of the X-ray spectrometer. No significant differences were noted in the diffraction pattern for a given composition of clinker burned in different equipment. Neither were there any detectable differences in diffraction pattern for a given composition of clinker as influenced by soft burning or hard burning in the rotary kiln. In Fig. 5 there are shown the diffraction patterns for clinkers of composition A and B, burned in the DTA furnace, hard-burned clinkers of compositions C, D, E, and F, burned in the rotary kiln, and the patterns for each of the fired raw materials (fired to 1300 C) used in the raw mixes for clinker compositions C, D, E, and F.

In Fig. 5 various parts of the patterns

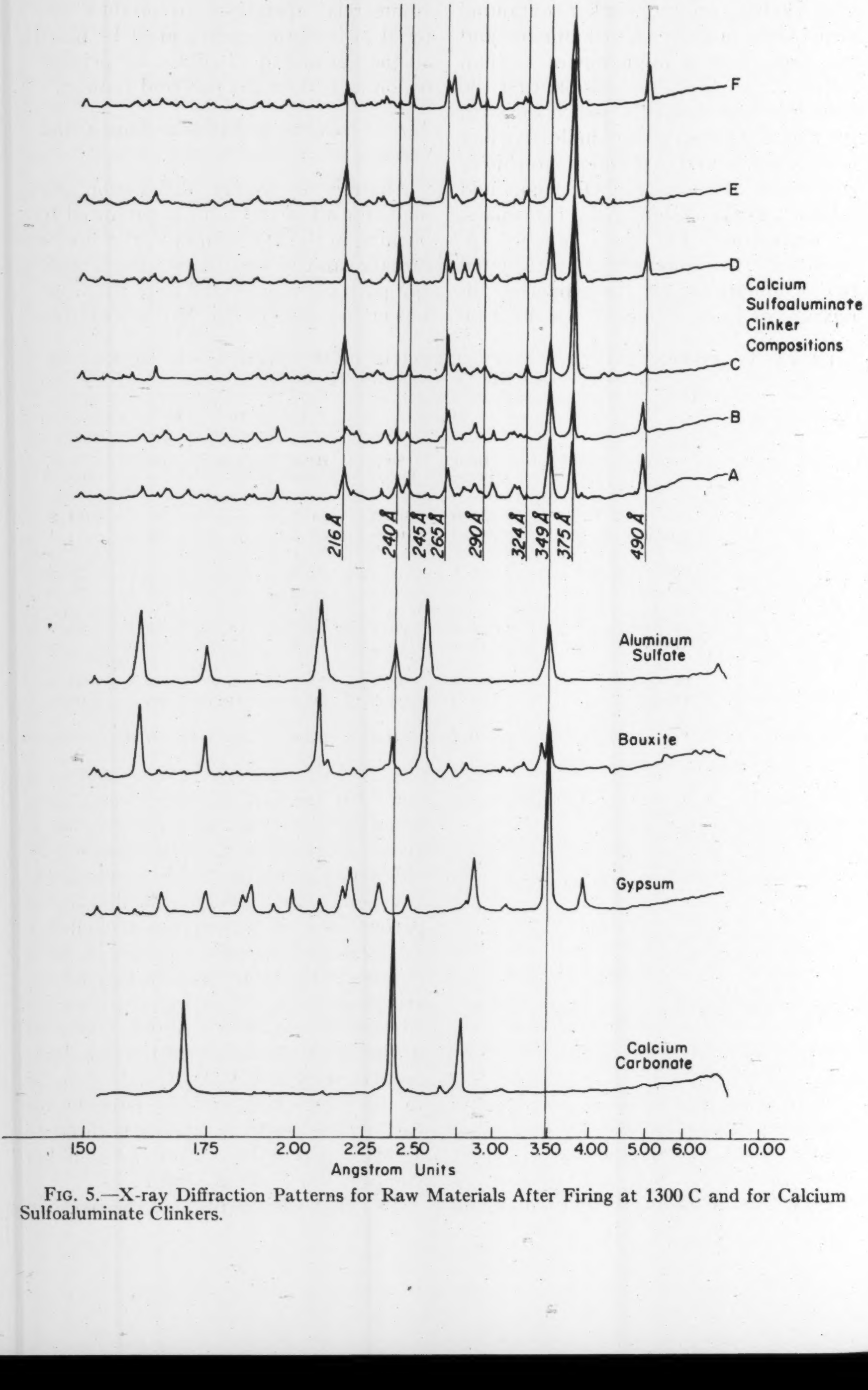

FIG. 5.—X-ray Diffraction Patterns for Raw Materials After Firing at 1300 C and for Calcium Sulfoaluminate Clinkers.

are designated by a reference line for which the value of the interplanar spacing, *d*, is shown. In general, the diffraction patterns are quite similar for the six clinker compositions. Examination of the patterns for spacings less than 2.0 Å indicated that compositions A and B are significantly different from the remaining clinker compositions. The patterns for clinkers C and E appear identical, while those for clinkers D and F are different from each other and from the other clinkers as well. Examination of the patterns for spacings greater than 2.0 Å indicated that all six clinkers are nearly alike in composition. In this range, some of the highest intensity lines in the patterns for the fired raw materials coincide with the pattern for some of the clinkers. For example, the maximum intensity line at 2.40 Å in fired calcium carbonate, that is CaO, is missing in the patterns for clinkers C and E, is of high intensity in the pattern for clinker D, and though present it is of weak intensity in the pattern for clinker F. It will be recalled that the chemical analyses of the clinkers (Table IV) indicated no free lime in clinkers C and E, about 21 per cent free lime in clinker D, and about 5 per cent free lime in clinker F.

TABLE VI.—X-RAY DIFFRACTION SPECTRA ATTRIBUTED TO COMPOUND $4CaO \cdot Al_2O_3 \cdot SO_3$.

Interplanar Spacing, *d*, Å	Relative Intensity
3.75	100
3.49	30
2.65	30
2.16	30
2.45	10
3.24	Less than 10
2.90	Less than 10
4.90	Less than 10
4.31	Less than 10
3.14	Less than 10

For fired gypsum, bauxite, and aluminum sulfate, a line of high intensity occurs at 3.49 Å. The same line appears at high intensity in each of the patterns for the six clinkers. Therefore, based on this single value in the X-ray patterns, it cannot be deduced whether this line of the pattern is characteristic of Al_2O_3, $CaSO_4$, or of the sulfoaluminate clinker. However, subsequent petrographic examinations showed that free Al_2O_3 is not present in any of the clinkers.

After checking all spectra which could be accounted for by compounds listed in available X-ray literature, it was found that at least nine lines in the patterns for the clinkers remained. These are listed in Table VI in the order of their relative intensities. The three lines of maximum intensity in the clinker patterns are at 3.75, 2.65, and 2.16 Å, in order of relative intensity. Examination of the data indicated that the line at 3.49 Å is also present in the pattern of the compound believed to be an anhydrous calcium sulfoaluminate, with a relative intensity of about 30, allowing for some reinforced intensity due to the presence of small amounts of calcium sulfate.

TABLE VII.—ESTIMATED COMPOUND COMPOSITION OF HARD BURNED CLINKERS BASED UPON OXIDE COMPOSITION AND X-RAY DIFFRACTION DATA.

Percentages by weight.

	Clinker C	Clinker D	Clinker E	Clinker F
Calcium sulfoaluminate	75	65	80	70
C_3A	10	5	0	5
$CaSO_4$	10	5	15	15
Free CaO	0	20	0	5
Miscellaneous impurities	5	5	5	5

Based upon a study of the X-ray data (conceded to be of limited value for quantitative purposes), particularly with respect to relative values for the line of maximum intensity of each compound, and taking into consideration the oxide analyses of the clinkers, the compound composition was estimated for clinkers C, D, E, and F as shown in Table VII. The principal compound in each clinker was calculated to be a calcium sulfoaluminate anhydrite, present in amounts ranging from 65 to 80 per cent in the four clinkers.

Based solely upon the oxide compositions of rotary-kiln products, as shown in Table IV, calculations were made of the probable composition of the calcium sulfoaluminates present, correcting for contents of free CaO and impurities. Whereas the raw mixes were designed on the basis of a composition of tetracalcium sulfoaluminate ($C_3A \cdot CaSO_4$), the oxide compositions remaining after correction for impurities and free CaO are fairly close to the following:

For compositions C and E:

$$5CaO \cdot 2Al_2O_3 \cdot SO_3$$

which can be written

$$C_3A \cdot CA \cdot CaSO_4 \ldots\ldots\ldots\ldots (1)$$

and for composition D:

$$9CaO \cdot 4Al_2O_3 \cdot 3SO_3$$

which can written

$$C_3A \cdot 3CA \cdot 3CaSO_4 \ \ldots\ldots\ldots (2)$$

and for composition F, a mixture of the compounds 1 and 2 and of a considerable amount of $CaSO_4$. Obviously a study of the ternary system $CaO \cdot Al_2O_3 \cdot SO_3$, together with X-ray analysis of thermal reaction products, is required to determine the true compound composition of the rotary-kiln products. It should be noted that for the theoretical compounds computed, composition D is significantly higher in SO_3 than are compositions C and E, and the high free CaO content of composition D could be the result of improper raw mix design for the manufacture of a potential $C_3A \cdot 3CA \cdot 3CaSO_4$. It may be noted also that the theoretical compounds 1 and 2 bear some similarity to the low sulfate and high sulfate hydrates $C_3A \cdot CaSO_4 \cdot 12H_2O$ and $C_3A \cdot 3CaSO_4 \cdot 30$ to $32H_2O$ respectively, the high sulfate being the most expansive hydrate.

Petrographic Description:

Petrographic examinations of the six clinkers were made by the U. S. Bureau of Reclamation in order to estimate the composition of the clinkers and the optical properties of the major constituent of calcium sulfoaluminate anhydrite. Oil immersion and polished thin section techniques were employed, requiring high magnification (970 ×). Inasmuch as the samples of the different clinkers, both hard burned and soft burned, differed only in the amounts of constituents, they were described together as follows:

The texture consisted of a mosaic of euhedral to anhedral crystals of microscopic size (0.01 to 0.015 mm) in a glass matrix. Slightly larger crystals appeared in zones throughout the clinker indicating nonuniform cooling of the melt. The predominant mineral appeared as colorless euhedral crystals which consisted of isotropic cubes and hexagonal plates with an index between 1.58 and 1.59. Colorless anhedral grains having an index of 1.58 to 1.59 and low birefringence were observed in small amounts. This material was considered to be the mineral anhydrite (calcium sulfate). The glassy material found in moderate amounts consisted of amber-colored, anhedral isotropic grains with a variable index of about 1.72, and a small amount of rounded grains with an index greater than 1.72, considered to be CaO.

A petrographic analysis by the Field Research Section of the Portland Cement Assn. gave the composition as consisting predominantly of a mineral of heretofore unknown X-ray and optical properties, small amounts of high-temperature calcium sulfate, small amounts of C_3A, and, in some of the clinkers, free CaO. This partly confirmed the estimated compound composition as given in Table VII.

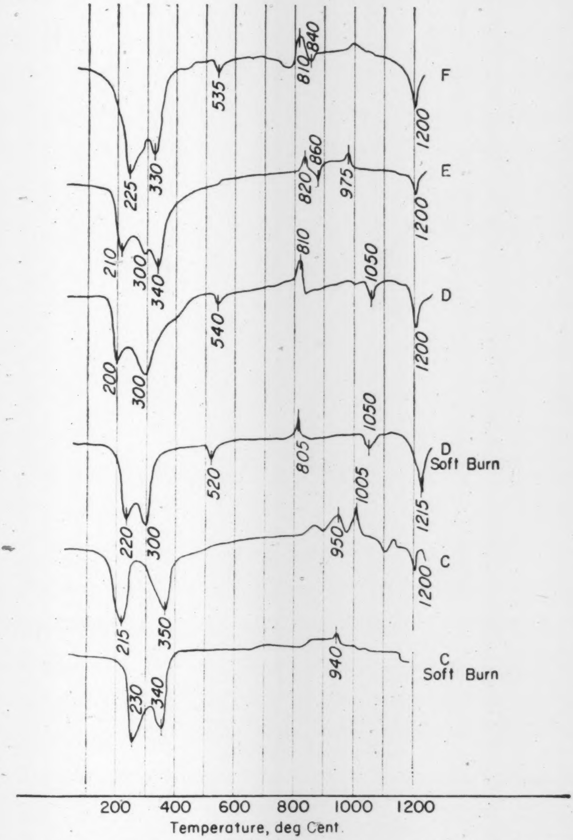

FIG. 6.—Differential Thermal Analysis Curves for Calcium Sulfoaluminate Clinkers Hydrated at 20 C.

HYDRATES OF SULFOALUMINATE CLINKERS

Samples of soft-burned and of hard-burned clinkers of compositions C and D, and hard-burned clinkers of compositions E and F were ground to about equal fineness (approximately 4000 sq cm per g by air-permeability apparatus) and then hydrated. Water in the amount of 70 per cent by weight of ground clinker was used in all hydration tests. The pastes for each condition were cast in 2 by 4-in. cylinder molds and kept at 20 C for 24 hr before removal of the molds. A complete group of six hydrates was thereafter stored in water at 20 C to age 7 days, then dried at 20 C and 50 per cent relative humidity to age 14 days. A corresponding group of hydrates was stored in water at 80 C from age 1 to 7 days, then dried at 20 C and 50 per cent relative humidity to age 14 days. The specimens

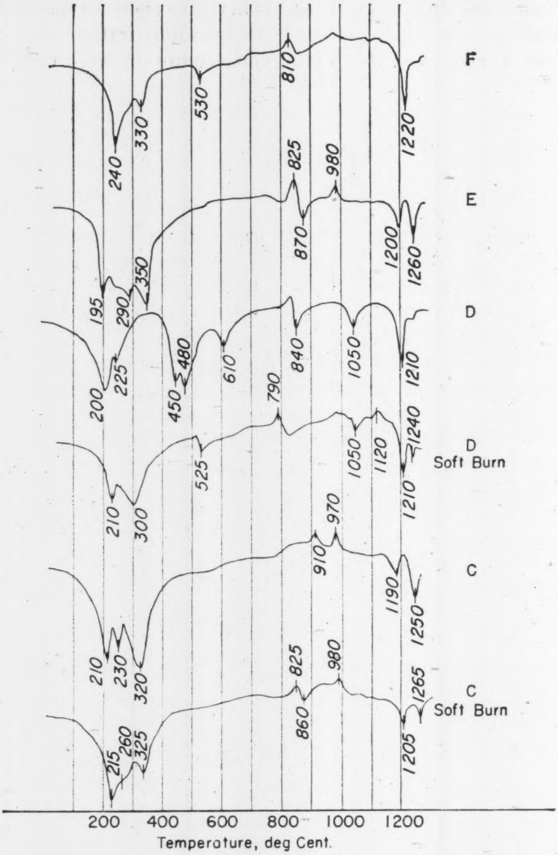

FIG. 7.—Differential Thermal Analysis Curves for Calcium Sulfoaluminate Clinkers Hydrated at 80 C.

were then ground for use in studies of thermal decomposition, differential thermal analysis, and analysis by X-ray diffraction.

Differential Thermal Analysis of Hydrates:

Differential thermal analyses were made for each of the twelve hydrates obtained by hydrating samples of each clinker at 20 C, and corresponding samples at 80 C. In Fig. 6 there are shown the thermal curves for ground clinkers hydrated at 20 C and in Fig. 7 there are shown curves for corresponding clinkers hydrated at 80 C. Above 700 C the exothermic and endothermic peaks probably are the result of recombinations after dehydration, and indicate the same inversions as noted in the thermal curves for raw mixes. There do not appear to be significant differences between hydrates obtained at 20 C as compared to hydrates obtained at 80 C. The 80 C hydrates for

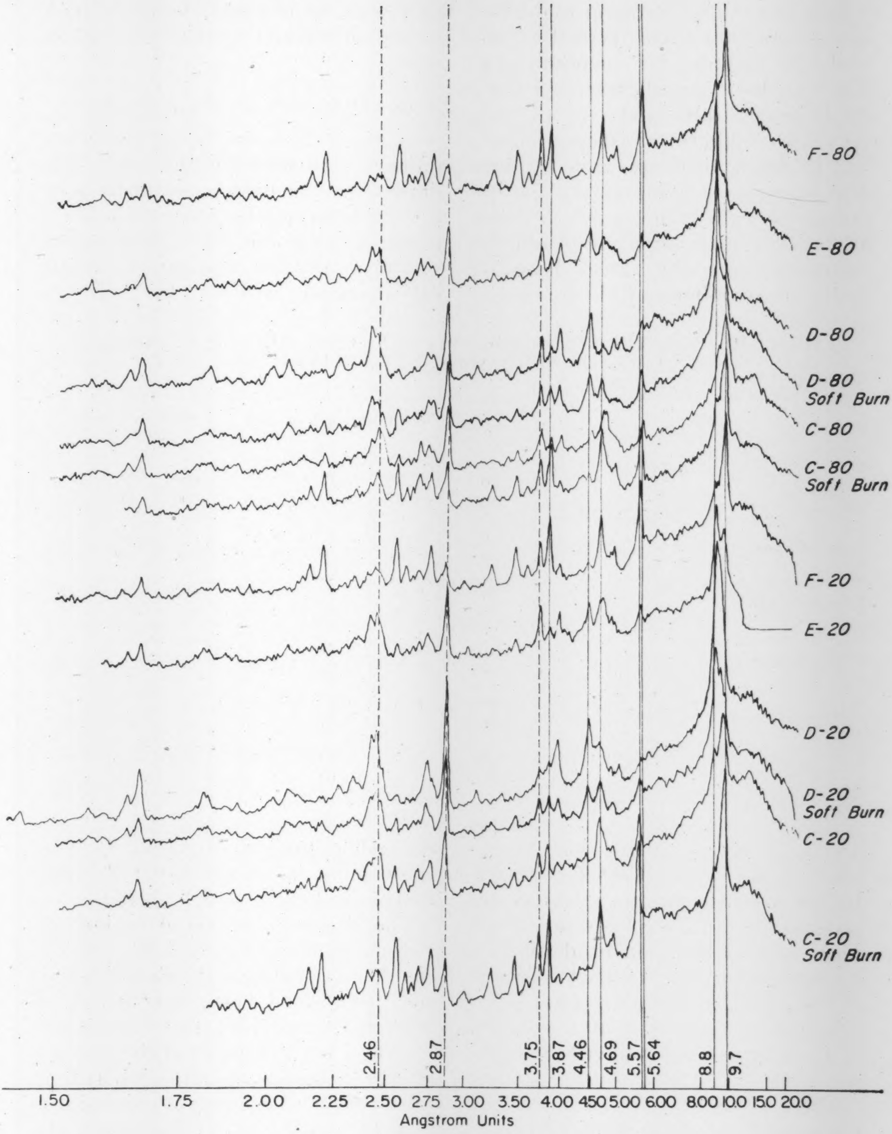

Fig. 8.—X-ray Diffraction Patterns for Hydrates of Calcium Sulfoaluminate Clinkers.

compositions C and E appear to produce substantially the same thermal curves, while the hydrates for compositions D and F produce thermal curves which differ from each other and also from the curves for hydrates of compositions C and E. Again, there appear to be three distinct types of hydrates. The optical properties of the hydrates, not available at present, may serve to distinguish clearly between the three classes of hydrates. According to F. E. Jones (in a private communication), his "recent work on the system $CaO-Al_2O_3-CaSO_4-H_2O$ at 25 C indicates that three modifications of $C_3A \cdot CaSO_4$ plus water occur in the aqueous system, since in addition to a longest basal spacing of 8.9 Å, normally attributed to $C_3A \cdot CaSO_4$ plus water, longest basal spacings of 10.3 Å and 9.6 Å have also been observed in 'moist solids.' "

TABLE VIII.—X-RAY DIFFRACTION SPECTRA FOR HYDRATES OF GROUND CALCIUM SULFOALUMINATE CLINKERS.

Clinker	Temperature of Hydration, deg Cent	Interplanar Spacing, *d*, Å			Relative Intensity		
C (soft-burn)	20	9.6	5.57	3.87	100	70	60
	80	9.6	8.8	4.69	100	60	60
C (hard-burn)	20	9.6	2.87	8.8	100	90	60
	80	9.6	2.87	9.0	100	80	70
D (soft-burn)	20	9.3	2.87	8.9	100	80	50
	80	8.9	9.6	2.87	100	70	60
D (hard-burn)	20	9.0	2.87	2.45	100	70	40
	80	8.9	2.87	4.46	100	50	30
E (hard-burn)	20	8.9	9.6	2.87	100	70	70
	80	8.9	2.87	4.46	100	50	30
F (hard-burn)	20	9.6	8.8	4.69	100	60	60
	80	9.7	5.64	4.69	100	80	60

There is no evidence in any of the curves of the presence either of hydrated calcium sulfate or of hydrous alumina. If calcium hydroxide was present in any of the hydrates, the amount was insufficient to register the normal endothermic peak typical of calcium hydroxide at about 560 C.

X-Ray Diffraction Studies of Hydrates:

In Fig. 8 there are shown the X-ray diffraction patterns for the twelve hydrates. The solid lines normal to the scale of interplanar spacings indicate the interplanar spacings which it is believed are typical of the whole group of calcium sulfoaluminate hydrates. These are, in descending order of magnitude in Ångstrom units, 9.7; 9.6; 9.3; 9.0; 8.9; 8.8; 5.64; 5.57; 4.69; 4.46; 3.87; (2.87).

The X-ray diffraction data indicate that no calcium sulfate hydrate occurs in the 20 C hydrates, and only a trace of such is indicated in the 80 C hydrate of the hard-burned clinker of composition D. There are indications of the presence of very small amounts of calcium hydroxide for the 20 C hydrates of compositions C and F, and in the 80 C hydrates of the soft-burned composition C. There is no evidence of calcium hydroxide in any of the hydrates of composition D. This is somewhat surprising when it is recalled that the free CaO content of clinker of composition D was nearly 20 per cent.

In Table VIII there are summarized the X-ray diffraction spectra for the several hydrates, showing only the three lines in each pattern having the highest intensities. The lines of maximum intensity agree well with values of longest basal spacings quoted by Jones. However, the available data from X-ray diffraction, differential thermal analysis, and thermal decomposition studies are insufficient and inadequate for purposes of determining the molar proportions of water in the various hydrates.

TABLE IX.—VOLUME CHANGES OF 1:2.75 CEMENT MORTARS CONTAINING CALCIUM SULFOALUMINATE ADMIXTURES.[a]

Condition	Portland Cement,[b] per cent	Expansive Admixture[c]			Soaking Time, days	Length Change, millionths per unit							
		Specific Surface[d]				Soaking, days			Drying				
		Designation	sq cm per g	Amount, per cent		1	6	120	1 day	7 days	28 days	4 months	9 months
No. 1........	100	...	...	...	6	12	38	...	−34	−399	−665	−800	−908
No. 2........	73	DS[e]	2950	22	6	2275	2689	...	...	2056	1789	1798	1982
No. 3........	73	DS	3750	22	6	1658	1977	...	...	1298	971	873	883
No. 4........	73	DS	4150	22	6	1275	1500	...	...	858	551	452	430
No. 5........	73	DS	4150	22	120	1002	1262	1650	1415	838	435	331	309
No. 6........	73	DH[f]	3200	22	6	11523	11692	...	...	11086	10534	10412	10294
No. 7........	73	DH	3800	22	6	8046	8246	...	...	7523	7077	6851	6770
No. 8........	73	DH	4300	22	6	6815	6973	...	...	6216	5786	5540	5453
No. 9........	73	DH	4300	22	120	6333	6572	6790	6562	5537	5370	...	5300

[a] Bars 1 by 1 by 10 in.; water-cement ratio, 0.50 by weight.

[b] Portland cement No. 1.

[c] Five per cent slag in all blended cements.

[d] Blaine fineness.

[e] DS = composition D, soft burned.

[f] DH = composition D, hard burned.

It appears, considering only the hard-burned clinkers, that three principal hydrates were involved—a hydrate of low water content for composition F, a hydrate of higher water content for compositions C and E, and a hydrate of highest water content for composition D. It will be recalled that the X-ray diffraction data for the clinkers (Fig. 5) and the oxide compositions (Table IV) also indicated three basic clinkers of different characteristics, that is, C and E, D, and F. It appears at this point that the structure of the hydrates obtained is dependent upon, and can probably be controlled by, the composition of the raw mix for manufacture of sulfoaluminate clinker and upon the control of the burning process.

MANUFACTURE OF EXPANSIVE CEMENTS

Each of the six sulfoaluminate clinkers was ground to each of three finenesses approximating specific surfaces of 3000, 3800, and 4200 sq cm per g as measured by the Blaine apparatus. Blended cements were then prepared using 22 per cent of the sulfoaluminate admixture, 5 per cent blast-furnace slag ground to a Blaine fineness of 4560 sq cm per g and 73 per cent of portland cement. The chemical analyses of the two commercial portland cements used are shown in Table II. These two cements, which differed appreciably in their analyses, were selected so as to evaluate the influence of such differences on their suitability for use in expansive cements.

Volume Change Studies:

To evaluate the expansive characteristics of these cements in comparison with those for the cements without any admixtures, 1 by 1 by 10-in. mortar bars were prepared using a 1:2.75 mix by weight with graded Ottawa sand, and a water-cement ratio of 0.50 by weight. At age 1 day they were immersed in water at 70 F. Some were water-soaked 6 days while others were soaked 4 months. Thereafter they were stored in air at 70 F and 50 per cent relative humidity. Length changes were observed during both the soaking and drying periods.

Test results for the admixture of composition D in both the soft-burned and the hard-burned condition are shown in Table IX. All these data are for the mixes using portland cement No. 1, as it was noted that none of the mixes using portland cement No. 2 were significantly expansive, due possibly to its high alkali and high C_3A contents. The bars containing no expansive admixture (condition No. 1) acted normally. They expanded 38 millionths per unit length after soaking 6 days and had a net contraction of 900 millionths after drying 9 months. In contrast thereto, the bars containing the soft-burned clinker DS (conditions 2 to 4) expanded 1500 to 2700 millionths per unit length while soaking 6 days and retained an expansion of 400 to 2000 millionths after drying 9 months, so that all possibility of shrinkage cracking should be eliminated. The expansion caused by the coarse admixture during 6 days soaking was about double that caused by the fine admixture, and after drying 9 months the final net expansion for the coarse admixture was about five times that for the fine admixture. For condition 2, some additional expansion occurred even after drying for 6 months. The initial and final expansions resulting from soaking for 120 days (condition 5) did not differ much from those for the 6-day soaking period (condition 4).

For the hard-burned clinker DH, the expansions while soaking 6 days ranged from 7000 to 12,000 millionths per unit length and after drying 9 months the bars still retained a major part of the initial expansion, so that these cements have potential use for self-stressing purposes. Again, the coarse admixture developed about double the expansion shown by the fine admixture, and the expansion resulting from the 120-day soaking did not differ much from that for the 6-day soaking, or even the 1-day soaking.

When the percentage of expansive admixture ranged from 10 to 20 to 30 per cent of the total cementing material, it was noted that the resulting expansions increased, for all ages, with increase in the amount of the admixture. This indicated that the amount of the expansion could be controlled within limits by adjusting the amount of the admixture used in the expansive cement.

After some of the satisfactory expansive D admixture had been thoroughly exposed in thin layers to the air in the laboratory for one week, during the season of high atmospheric humidity, it was found to have lost much of its expansive properties, showing the need for care in storing such cements.

The Lossier cement obtained from France did not develop expansions which are normally expected for it, but this may have resulted from deterioration caused by aging.

In general, clinkers C, E, and F did not exhibit satisfactory expansive characteristics. It is believed that this resulted either from the low free lime content of these clinkers or the absence of the appropriate calcium sulfoaluminate compound which did not permit the necessary expansive reactions to occur.

Compressive Strength:

The compressive strength for 2 by 4-in. cylinders of 1:2.75 mortars with a water-cement ratio of 0.55 by weight was determined. The specimens were cured in water at 70 F to age 7 days and thereafter stored in air, as for the volume change bars. The mortar for the cement No. 1 with 20 per cent of the soft-burned admixture D of low fineness developed a compressive strength of 3680 psi at age 14 days and that for the admixture of high fineness developed a strength of 4405 psi. The corresponding value for the mortar with the hard-burned admixture D of high fineness was 3500 psi and for the cement No. 1 without any sulfoaluminate mixture, but with 5 per cent of slag as for the other specimens, the compressive strength was 3860 psi. The mortar cylinders for cement No. 2, without any sulfoaluminate admixture, developed a compressive strength of 4820 psi at 28 days, while for the same cement with the hard-burned admixture D of low fineness the corresponding strength was 4650 psi.

The strength values for the mortars containing admixture D show that this material is probably capable of serving satisfactorily in structural concrete.

CONCLUSIONS

The following conclusions may be drawn from the investigation:

1. It is possible to design a raw mix from commercial CaO-Al_2O_3-SO_3 materials which, when burned to incipient fusion or even slightly below, produces a clinker which is predominantly an anhydrous calcium sulfoaluminate compound.

2. Although this compound could not be identified by differential thermal, X-ray diffraction, or petrographic analyses, due to lack of published data on the characteristics of such a compound, these analyses pointed to the possibility of its being a calcium sulfoaluminate of unknown compound composition.

3. A clinker of this type having in its oxide composition a total CaO content of about 53 per cent and a free CaO content of about 19 per cent, when ground and used in a blended cement having the proportions 20:5:75 of sulfoaluminate, slag, and portland cement had definite expansive characteristics.

4. An expansive cement of the above proportions, prepared from a soft-burned sulfoaluminate clinker and used in 1 by 1 by 10-in. mortar bars of a 1:2.75 mix, water cured 6 days, and then air dried 9 months may have a net expansion of 400 millionths per unit for a finely ground admixture or 2000 millionths per unit for a somewhat coarser admixture. Corresponding expansions in concrete (say one third of these values) could prevent formation of shrinkage cracks in a concrete structure.

5. For a corresponding cement prepared from a hard-burned clinker, the net expansions of the mortar bars after air drying 9 months may be 5000 and 10,000 millionths per unit respectively for the fine and coarse admixtures. The corresponding smaller expansions for concrete may be sufficient for producing self-stressed concrete.

6. For sulfoaluminate clinkers of about the same composition as in 3 above, but containing little or no free CaO, the corresponding blended cements had no expansive characteristics and even produced excessive shrinkage.

7. Portland cements high in alkalies and C_3A did not produce expansive cements even when blended with these

potentially expansive sulfoaluminate admixtures.

8. Expansive cements of the same composition as in 3 above developed compressive strengths satisfactory for structural concrete.

Acknowledgments:

The authors gratefully acknowledge the assistance of the U. S. Bureau of Reclamation and the Portland Cement Assn. in providing the petrographic analyses of the sulfoaluminate clinkers.

They also acknowledge the valuable assistance of Marjorie Kelso, Division of Mineral Technology, and Louis Trescony, Division of Civil Engineering, University of California, in the conduct and interpretation of X-ray and differential thermal analyses.

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DISCUSSION

MR. WALTER H. PRICE.[1]—About four weeks ago I visited the laboratory of V. V. Mikhailov in Moscow, and found that the Russians have about 25 people working on the product they call self-stressing cement. They claim that by controlling the proportions of the ingredients, the fineness of the product and methods of curing they can control the expansion. I understand that at present the Russians have nothing that we do not know, but they are putting a lot more effort than we are into this particular endeavor.

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VARIATIONS IN PORTLAND CEMENT*

By Stanton Walker and Delmar L. Bloem[1]

Much ready-mixed concrete is sold with strength as the basis for acceptance. Control of strength is complicated by many factors. Variations due to processing, handling and testing may be minimized by the producer in cooperation with the user, but those due to the ingredients present a more difficult problem.

Lack of uniformity in portland cement is an important cause of non-uniform concrete strengths. That there are large differences in the strength-producing properties of cements from different sources is well known, although not as generally taken into account in concrete control as it should be. That significant differences, above specification minima, occur for different shipments from the same mill has not been generally recognized.

In a study of the cement variable, particularly as represented by different shipments from a given source, tests were conducted in the Joint Research Laboratory of the National Sand and Gravel Assn. and the National Ready Mixed Concrete Assn. at the University of Maryland. Samples were collected over a period of one year. Both mortar and concrete tests were made.

Scope of Investigation

Samples were secured from each of five sources every two weeks from October, 1955, to October, 1956. A sixth cement consisting of a blend of five brands from the Washington area was thoroughly mixed and stored in sealed containers for use as a standard of comparison.

The samples were furnished by member companies selected for geographical reasons and as having competent engineering personnel to supervise their collection. They were shipped to the University of Maryland in sealed, waterproof-paper-lined cloth bags, having a capacity of about 35 lb of cement. The shipping dates were arranged so that samples from the several sources arrived at the laboratory on or about the same date. When a sample was received, it was prepared as specified in ASTM Method of Test for Compressive Strength of Hydraulic Cement Mortars (Using 2-in. Cube Specimens) (C 109).[2] Portions were weighed out in proper quantities for individual batches and then placed in sealed Mason jars. At the same time, two 1-gal containers were filled, sealed and set aside for later tests.

Three principal series of tests were conducted at different times. The first involved standard mortar tests on all

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* Presented at the Sixty-first Annual Meeting of the Society, June 22–27, 1958.

[1] Director and Associate Director of Engineering, respectively, National Sand and Gravel Assn. and the National Ready Mixed Concrete Assn., Washington, D. C.

[2] 1956 Supplement to ASTM Book of Standards, Part 3, p. 129.

TABLE I.—SUMMARY OF COMPRESSIVE STRENGTH TESTS OF MORTAR (SERIES 158).

Each value average for 15 cubes; 3 from each of 5 batches mixed on different days, made and tested in accordance with ASTM Method C 109. Source No. 1 was the control sample, a blend of 5 brands, with the 26 portions sampled and sealed at the same time. s_1, s_2 and s_3 are, respectively, standard deviations in psi, within batch, batch-to-batch and sample-to-sample; v_1, v_2 and v_3 are corresponding coefficients of variation.

	Compressive Strength, psi, for Cement Samples from 6 Sources																	
Sample	3-Day Tests						7-Day Tests						28-Day Tests					
	1	2	3	4	5	6	1	2	3	4	5	6	1	2	3	4	5	6
No. 1	2651	2959	1965	1899	2709	1598	3897	4067	3125	2486	4201	2475	5357	5254	4937	3364	5785	4126
No. 2	2581	2851	2156	1892	3005	2143	3729	3845	3476	2451	4426	3197	5216	4809	5295	3453	6049	4892
No. 3	2385	2755	....	2030	2588	1731	3608	4061	....	2641	3978	2691	5211	5445	....	3546	5684	4538
No. 4	2575	2295	....	2421	2680	2061	3794	3443	....	2999	4205	3028	5181	4820	....	3909	5920	4692
No. 5	2394	2196	2159	1907	2759	1699	3548	3232	3380	2536	4083	2599	5089	4628	5116	3511	5668	4422
No. 6	2432	2454	2048	2099	2673	2082	3643	3853	3349	2733	3954	3172	5235	5217	5117	3643	5313	4883
No. 7	2382	2514	2254	1756	2889	1925	3619	3828	3616	2422	4150	2983	5115	5076	5417	3355	5837	4728
No. 8	2369	2716	2143	2215	2538	1670	3597	4134	3472	2873	3639	2556	5175	5429	5327	3833	5410	4372
No. 9	2322	2366	1995	2323	2783	1591	3660	3721	3068	3112	3819	2432	5128	5143	4934	4099	5262	4314
No. 10	2440	2778	2306	2120	2906	1966	3729	4143	3544	2684	4150	2993	5258	5510	5378	3635	6042	4758
No. 11	2478	2637	2385	1925	2696	2165	3791	4164	3675	2614	4090	3083	5259	5299	5519	3689	5693	4748
No. 12	2518	2511	2331	2055	2726	2072	3734	3815	3777	2810	4196	3088	5353	5062	5518	3775	5936	4659
No. 13	2535	3035	2061	2314	2576	2215	3780	4345	3376	2768	4030	3185	5412	5518	5045	3663	5675	4855
No. 14	2451	3363	1980	1841	2500	1570	3595	4813	3147	2376	3603	2385	5083	5900	4608	3271	5098	3923
No. 15	2528	3339	1868	2525	2666	1998	3780	5026	3041	3472	4110	2967	5072	6044	4833	4257	5582	4741
No. 16	2481	2818	1961	1744	2642	2023	3615	4295	3066	2477	4053	3053	5097	5542	4804	3485	5670	4726
No. 17	2477	3198	1963	2081	2541	1740	3500	4446	3056	2552	3624	2641	5225	5751	4932	3427	5605	4500
No. 18	2443	2889	1854	2003	2459	1960	3559	4070	2903	2877	3372	2913	5157	5556	4808	4132	4949	4640
No. 19	2484	2899	2124	1910	2130	1968	3655	4150	3222	2488	3080	2969	5246	5471	5019	3541	4838	4784
No. 20	2465	....	....	2026	2342	2035	3718	....	....	2693	3481	3018	5236	....	....	3537	5311	4393
No. 21	2446	....	1852	2091	2615	1982	3720	....	3015	2707	3711	2961	5204	....	4900	3737	5514	4874
No. 22	2585	3043	1735	2050	2814	2060	3751	4374	2684	2689	4188	3096	5236	5680	4268	3637	5815	4616
No. 23	2368	2721	1977	2067	2875	2074	3655	3956	3056	2698	4283	3037	4847	5075	4569	3599	5531	4428
No. 24	2475	3105	1820	2270	2553	2036	3561	4202	2789	2810	3384	2890	5068	5399	4651	3781	4873	4356
No. 25	2360	2450	1679	2109	1801	2019	3572	3833	2706	2773	2620	2890	5447	5434	4631	3847	4348	4590
No. 26	2280	2887	1556	1874	1596	....	3632	4413	2564	2691	2425	....	5245	5451	4216	3836	3868	....
Average	2458	2782	2007	2060	2579	1935	3671	4093	3179	2709	3802	2892	5198	5355	4950	3674	5434	4582
s_1	61	74	46	47	73	42	99	98	81	53	114	67	133	132	125	79	137	111
s_2	87	104	68	69	90	65	98	114	99	58	115	90	119	131	127	81	137	114
s_3	87	315	210	196	318	192	96	389	330	236	502	244	123	339	362	244	520	245
v_1	2.6	2.7	2.3	2.3	2.8	2.2	2.7	2.4	2.6	2.0	3.0	2.3	2.6	2.5	2.5	2.1	2.5	2.4
v_2	3.6	3.7	3.4	3.4	3.5	3.3	2.7	2.8	3.2	2.1	3.1	3.1	2.3	2.4	2.6	2.2	2.6	2.5
v_3	3.6	11.3	10.5	9.5	12.4	9.9	2.6	9.5	10.4	8.7	13.2	8.5	2.4	6.3	7.3	6.6	9.6	5.3

samples; the second, concrete tests on selected samples; and the third, concrete and mortar tests on selected samples.

TABLE II.—MIXING WATER REQUIREMENTS AND FLOW OF MORTAR (SERIES 158).

Tabulated values are mixing water in milliliters for standard 9-cube batch (with 740 g cement and 2035 g graded standard sand) and flow of mortar in per cent (25 drops of table in 15 sec). Each value average for 5 batches mixed on different days.

Sample	Cement Source Number											
	1		2		3		4		5		6	
	Water, ml	Flow, per cent	Water, ml	Flow, per cent	Water, ml	Flow, per cent	Water, ml	Flow, per cent	Water, ml	Flow, per cent	Water, ml	Flow, per cent
No. 1	355	107	358	110	358	110	355	106	359	108	380	110
No. 2	355	112	355	102	355	112	355	112	355	105	364	109
No. 3	355	113	355	111	...	...	355	110	355	110	366	109
No. 4	347	109	355	105	...	...	345	112	355	109	373	108
No. 5	350	106	359	107	355	112	355	107	359	109	375	106
No. 6	350	107	356	111	359	106	351	108	346	109	375	113
No. 7	355	111	355	109	352	110	355	112	355	108	375	110
No. 8	355	110	355	109	350	109	356	104	356	105	375	110
No. 9	355	110	354	108	354	110	359	107	349	112	375	108
No. 10	355	112	355	110	350	111	355	109	355	108	367	107
No. 11	354	107	361	106	350	111	353	112	353	106	374	105
No. 12	353	107	353	107	350	108	355	105	354	109	375	107
No. 13	355	110	355	107	350	107	355	111	354	110	365	107
No. 14	355	107	355	107	354	106	355	109	361	105	375	107
No. 15	353	108	350	110	350	110	352	107	352	107	372	112
No. 16	355	110	355	108	355	112	356	107	356	107	375	112
No. 17	354	111	355	108	353	111	353	108	363	112	368	112
No. 18	353	110	355	108	352	111	352	108	352	106	364	109
No. 19	355	109	355	107	355	113	355	112	365	109	363	112
No. 20	355	111	...	...	...	...	355	111	365	110	365	108
No. 21	353	111	...	...	349	112	355	109	356	111	361	111
No. 22	353	112	353	108	350	113	356	105	355	108	360	111
No. 23	355	112	355	107	355	108	356	105	355	109	359	108
No. 24	355	112	355	110	355	113	355	107	355	101	356	106
No. 25	355	113	355	111	359	108	355	108	374	107	360	111
No. 26	355	108	365	106	359	105	355	106	395	105	...	...
Average	354	110	356	108	353	110	354	108	358	108	369	109
Range	8	7	15	9	10	8	14	8	49	11	24	8
Per Cent Range	2.3	6.4	4.2	8.3	2.8	7.3	4.0	7.4	13.7	10.2	6.5	7.3

The first series (No. 158) was made to develop information on variations in strength as measured by ASTM Method C 109 – 56. Five rounds were made on different days with each cement sample, including a portion of the standard sample; each round, including all sources, was made on one day. Nine cubes were molded from each batch for tests in triplicate at 3, 7, and 28 days. Accordingly, each strength value is the average of 15 tests. This series involved the molding and testing of approximately 7000 2-in. mortar cubes. The last strength tests were made during the first week of November, 1956.

In the second series (No. 158C), concrete having a cement factor of $5\frac{1}{2}$ sacks per cu yd and a slump of 3 to 4 in., for test at 3, 7, and 28 days, was made using samples of cement which had pro-

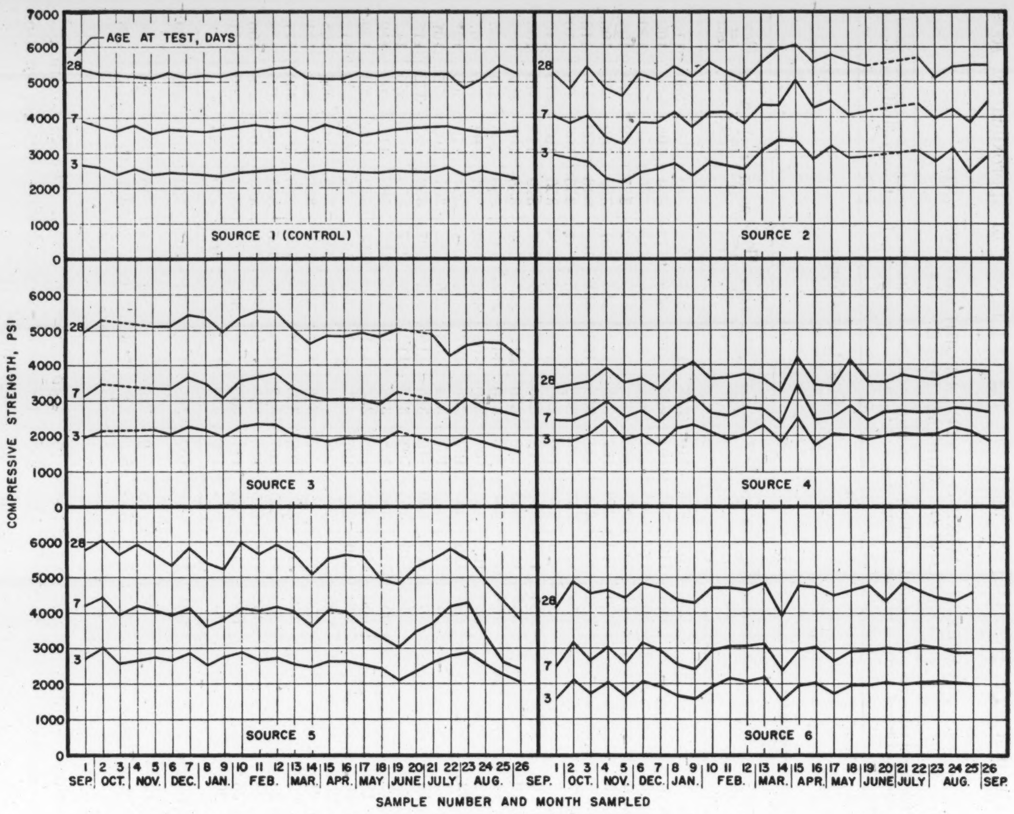

FIG. 1.—Compressive Strengths of Mortar for Repeated Sampling of Cement from Given Sources (Series 158).

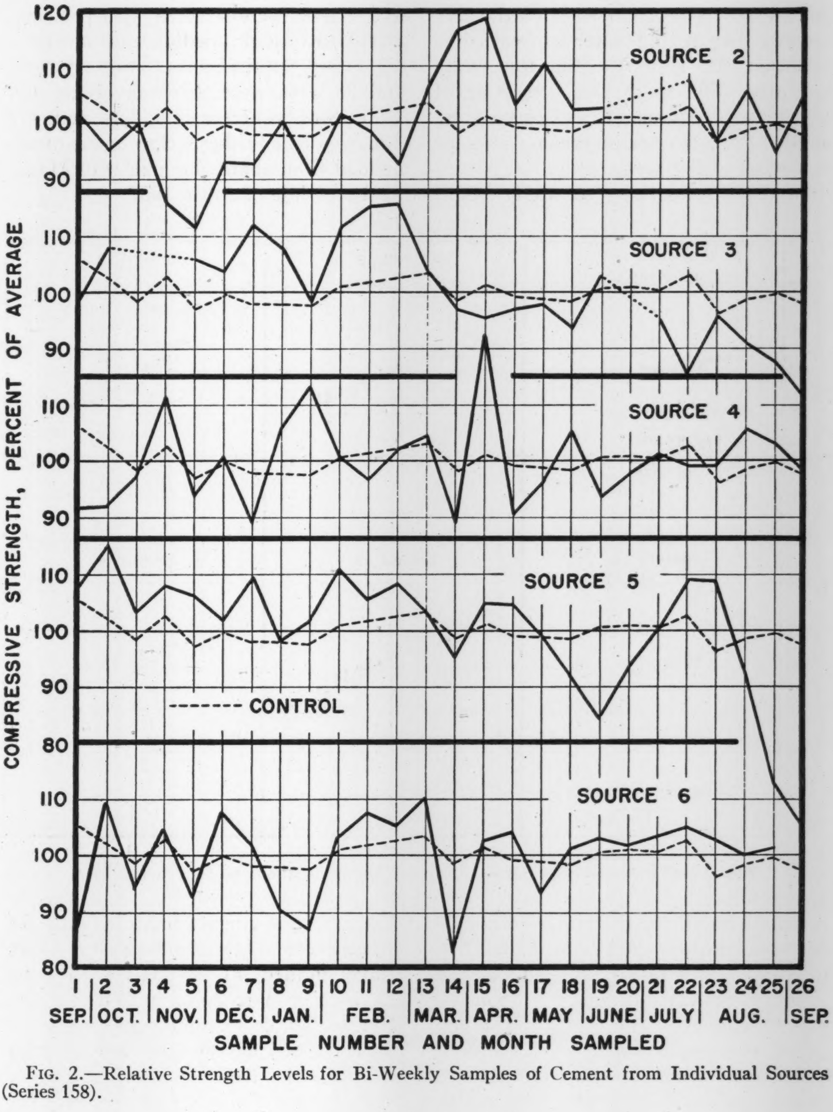

FIG. 2.—Relative Strength Levels for Bi-Weekly Samples of Cement from Individual Sources (Series 158).

duced the highest, median, and lowest mortar strengths from each of the five sources. The mixing water was varied to maintain the slump within the limits indicated. The specimens, consisting of 159 4 by 8-in. concrete cylinders, were molded the last week of January and the first week of February, 1957.

In the third series (No. 158D), both concrete and mortar tests were made, each in constant proportions and with constant water ratio, allowing the consistency to vary depending upon the water demand of the cement. The concrete was proportioned 1:2.72:3.95 by weight and contained about 5 sacks of cement per cubic yard. The mortar consisted of a 1:3.8 mix by weight of cement and graded standard sand. A water ratio of 0.65 by weight was used in both the concrete and mortar. For this series, three samples from each of the five sources were selected with principal attention to high, median, and low mixing water requirements. Tests at 3, 7, and 28 days were made involving 159 4- by 8-in. concrete cylinders and 477 2-in. mortar cubes. Specimens were molded from about the middle of March to the middle of April, 1957.

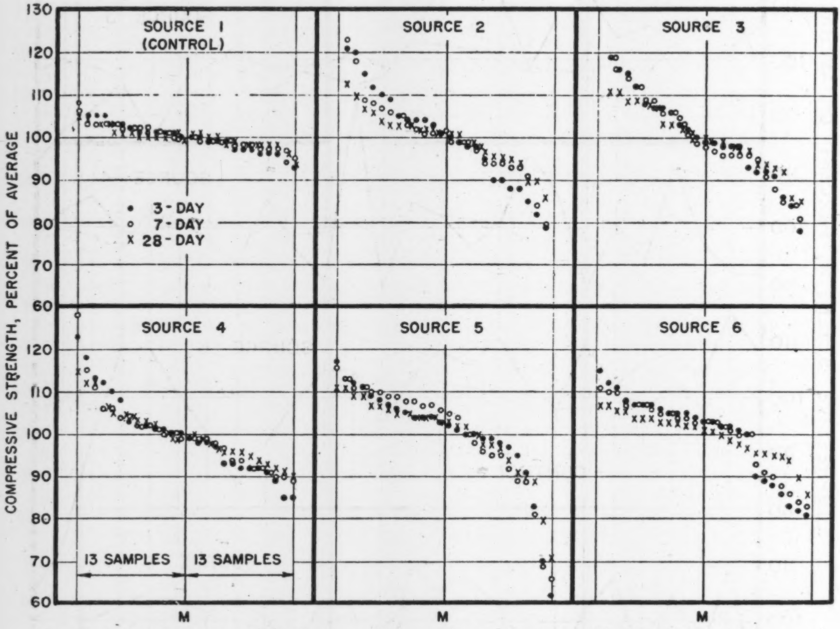

FIG. 3.—Percentage Distribution of Mortar Strengths About Median (Series 158).

The coarse aggregate used in both sets of concrete was a high-quality quartz gravel graded 40 per cent $\frac{3}{4}$ to $\frac{1}{2}$ in., 20 per cent $\frac{1}{2}$ to $\frac{3}{8}$ in., and 40 per cent $\frac{3}{8}$ in. to No. 4. The fine aggregate was a siliceous sand having a fineness modulus of 2.75. The concrete was hand-mixed in 0.25-cu ft batches which were tested for slump and unit weight before molding three cylinders for compressive strength tests at 3, 7, and 28 days. In both concrete series, three batches were made on different days for each cement sample tested. On each of the 8 days required to complete the concrete mixing for each series, a companion batch was made from the blended control cement. Procedures for mixing, molding and testing the concrete conformed to applicable ASTM standards.

Auxiliary tests were made to determine changes in flow with changes in mixing water and the effect of variations in grading of standard sand on the strength of ASTM C 109 mortar. Through the cooperation of the Portland Cement Assn., chemical analyses and fineness tests were made on 15 samples of cement, the three from each of the five sources used in series 158C. These represented the maximum, median, and minimum strength levels revealed by the standard mortar tests.

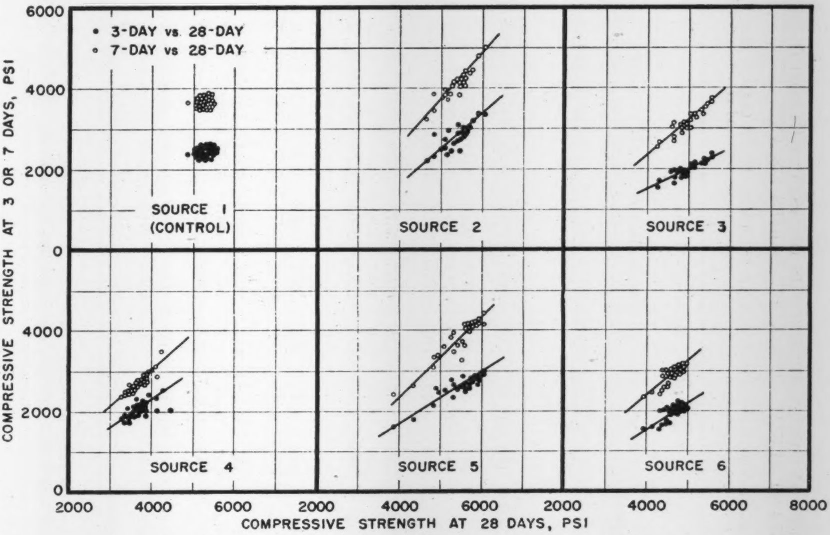

FIG. 4.—Relationships Between Strengths at 3 and 7 Days and Strengths at 28 Days (Series 158).

TEST RESULTS

This report gives the average results for the various test conditions. The individual data are too voluminous to publish here, but they are available to interested researchers on request.

ASTM C 109 Mortar Tests:

Table I gives the average strengths of the ASTM C 109 mortar for each of the bi-weekly samples from the five sources and the concurrent tests of the control samples. Standard deviations and coefficients of variation, accepted measures of uniformity, are also included. Table II gives the mixing water requirements and the consistency of the mortar as measured by the flow tests. The strength data are shown graphically in Figs. 1, 2 and 3.

The tables and diagrams indicate clearly that the cements from the five sources varied significantly from shipment to shipment. It is apparent that the ranges in strength for them are considerably higher than for the "control" (source 1), for which 26 tests on the same carefully prepared and preserved cement

TABLE III.—COMPARISONS BETWEEN STRENGTHS OF ASTM METHOD C 109 MORTAR AND CONSTANT SLUMP CONCRETE (SERIES 158 AND 158C).

Mortar strengths average of 15 cubes, 3 each from 5 batches, made and tested in accordance with ASTM Method C 109. Concrete strengths average of three 4 by 8 in. cylinders from separate batches, designed to contain 5.5 sacks of cement per cu yd with slump of 3 to 4 in.; aggregates quartz sand and gravel graded from 0 to 3/4 in.; sand 40.8 per cent total aggregate; combined fineness modulus 5.05.

Cement	Sample[a]	Mortar Strength Level[b]	C 109 Mortar						5.5-Sack Concrete						
			Water		Flow, per cent	Compressive Strength, psi			Water		Slump, in.	Weight, lb per cu ft	Compressive Strength, psi		
			ml[c]	Water-Cement Ratio by Weight		3 Days	7 Days	28 Days	Gal per cu yd	Gal per Sack			3 Days	7 Days	28 Days
Source 1[d]	All	..	354	0.48	110	2458	3671	5198	35.8	6.52	3.6	146.2	2200	3520	5010
Source 2	No. 15	High	350	0.47	110	3339	5026	6044	36.2	6.58	3.4	146.8	2655	4475	5360
	No. 8	Median	355	0.48	109	2716	4134	5429	36.9	6.72	3.2	146.8	2400	3955	5050
	No. 5	Low	359	0.49	107	2196	3232	4628	36.6	6.66	3.3	146.4	2055	3315	4665
Source 3	No. 11	High	350	0.47	111	2385	3675	5519	36.2	6.53	3.2	147.6	1915	3385	5070
	No. 9	Median	354	0.48	110	1995	3068	4934	36.8	6.65	3.2	147.7	1545	2765	4625
	No. 26	Low	359	0.49	105	1556	2564	4216	37.0	6.70	3.3	147.5	1415	2570	4370
Source 4	No. 15	High	352	0.48	107	2525	3472	4257	35.9	6.53	3.6	146.6	2195	3395	4250
	No. 22	Median	356	0.48	105	2050	2689	3637	36.5	6.62	3.2	147.1	1805	2700	3865
	No. 14	Low	355	0.48	109	1841	2376	3271	35.6	6.47	3.5	146.5	1660	2370	3365
Source 5	No. 2	High	355	0.48	105	3005	4426	6049	36.3	6.61	3.1	146.8	2375	3620	5185
	No. 15	Median	352	0.48	107	2666	4110	5582	36.1	6.57	3.5	146.5	2260	3675	5225
	No. 26	Low	395	0.54	105	1596	2425	3868	37.5	6.86	3.0	146.3	1705	2475	3980
Source 6	No. 2	High	364	0.49	109	2143	3197	4892	36.0	6.58	3.5	146.2	1595	2820	4470
	No. 18	Median	364	0.49	109	1960	2913	4640	36.3	6.62	3.3	146.5	1780	2875	4480
	No. 14	Low	375	0.51	107	1570	2385	3923	36.6	6.69	3.6	146.3	1490	2375	3915

[a] Samples from sources 2 through 6 taken at 2-week intervals for 1 yr, numbered consecutively.

[b] High, median and low-testing samples, based on averages of mortar strengths at 3 test ages.

[c] Average mixing water used with 740 g of cement and 2035 g of sand in 9-cube batch of mortar.

[d] Control sample prepared in advance and used as basis of comparison for each set of samples from outside sources. Mortar strengths are averages for all 26 sampling periods (total of 390 cubes for each age); concrete strengths are averages of 8 cylinders from separate batches mixed on different days.

were made. Figure 1 shows shipment-to-shipment variations in strength for the three ages, expressed in psi. In Fig. 2, the same data, averaged for the three test ages, are shown as percentages of the over-all average for each source. In Fig. 3, the percentage strengths at each age have been arranged in order of descending magnitude. In all three diagrams, the wider vertical distribution for the five sources than for the control is apparent.

TABLE IV.—COMPARISONS BETWEEN STRENGTHS OF MORTAR AND CONCRETE OF CONSTANT WATER-CEMENT RATIO (SERIES 158D).

Mortar strengths average of 9 cubes from 3 batches mixed and tested in accordance with ASTM Method C 109 except that proportions were fixed at 1 to 3.875 by weight with water-cement ratio of 0.65 by weight.

Concrete strengths average of three 4 by 8 in. cylinders from separate hand-mixed batches of fixed proportions, 1 to 2.72 to 3.95 by weight, with water-cement ratio of 0.65 by weight; aggregate quartz sand and gravel graded from 0 to 3/4 in.; sand 41.25 per cent total aggregate; combined fineness modulus 5.01.

Cement	Sample[a]	Mortar				Concrete					
		Flow, per cent[b]	Compressive Strength, psi			Water, gal per cu yd[c]	Slump, in.	Weight, lb per cu ft	Compressive Strength, psi		
			3 Days	7 Days	28 Days				3 Days	7 Days	28 Days
Source 1[d].	All	98	1033	1580	2537	37.0	5.6	146.0	1645	2830	4225
Source 2.	No. 14	99	1509	2302	3084	37.0	6.4	145.9	2105	3435	4480
	No. 26	92	1354	2072	2868	37.0	5.4	146.0	1850	3320	4415
	No. 2	98	1250	1837	2521	36.8	5.6	145.4	1735	2890	3815
Source 3.	No. 12	100	1091	1675	2781	37.2	4.3	146.9	1510	2730	4435
	No. 25	92	857	1356	2401	37.2	3.2	146.8	1095	2000	3630
	No. 22	103	720	1133	2019	36.9	5.3	145.8	1000	1800	3285
Source 4.	No. 4	98	968	1261	1991	37.2	6.2	146.6	1490	2080	2960
	No. 23	86	900	1316	2011	37.1	3.6	146.6	1350	1975	3070
	No. 7	90	726	1100	1757	37.0	4.0	146.0	1145	1800	2640
Source 5.	No. 10	91	1257	1805	2949	37.1	4.6	146.5	1745	2855	4370
	No. 6	102	1223	1798	2737	36.7	5.9	144.5	1910	3000	4315
	No. 25	76	951	1351	2148	37.3	2.8	147.2	1315	2085	3485
Source 6.	No. 6	83	1001	1500	2500	37.1	5.2	146.4	1440	2350	4005
	No. 24	91	949	1457	2327	37.0	4.7	145.8	1325	2170	3710
	No. 9	81	771	1168	2253	37.0	4.5	146.1	1095	1870	3635

[a] Same as samples tested in series 158; arranged for each source approximately in descending order of mortar strength in that series.

[b] For fixed quantity of mixing water (364 ml in 9-cube batch).

[c] Slight variation from design quantity of 37 gal per cu yd results from small differences in measured unit weight.

[d] Control sample; each value average for 8 batches which provided 24 cubes for each test age in the case of mortar and 8 cylinders for each age for concrete.

A more quantitative comparison of the strength variability can be derived from the values of standard deviation and coefficient of variation given in Table I. On the basis of sample-to-sample coefficients of variation, all five sources showed considerably poorer uniformity than the "control." These coefficients range from 7.9 to 11.7 per cent, as compared with only 2.9 per cent for the "control," when values for the three ages are averaged. Even for the lowest variation of any outside source, 7.9 per cent for No. 6, the likelihood of its greater variation than the control (2.9 per cent) being a coincidence, and not due to actually greater variability, is less than 1 in 1000.

Among the outside sources, No. 5 produced a significantly higher coefficient of variation than the other four. Differences in variability among sources 2, 3, 4, and 6 probably were not great enough to be significant. However, that the variations in strength shown for samples from these sources reflected actual differences in the cement is supported by the chemical analyses in Table VIII, discussed later.

In the case of cement No. 5, its higher coefficient of variation can be attributed entirely to the low strengths produced by the last two samples, Nos. 25 and 26. The sudden change in cement No. 5 is also reflected in the considerably greater mixing water required for mortar made with these two samples, as shown in Table II. The fact that the change did not occur earlier and that it was so sudden and so large may be considered more potentially damaging than if the cement had been "uniformly" variable.

Although the variability of mortar strength seems to decrease slightly with age at test, it appears that whatever characteristics of the cements caused the variations were active at all ages. This is demonstrated in Fig. 4 which shows the relationship between 28-day and 3- and 7-day strengths for the different sources. In all cases, except for the control, there is a good relationship among strengths

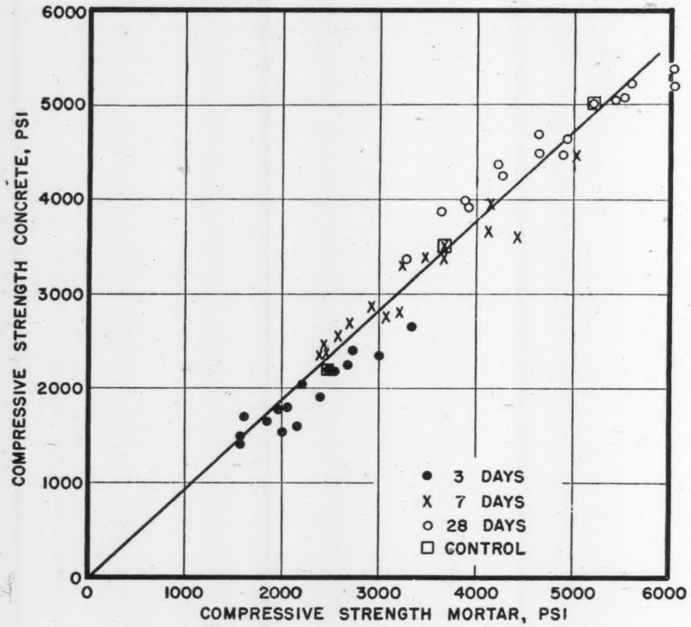

FIG. 5.—Relationship Between Compressive Strengths of Constant-Slump Concrete (Series 158 C) and C 109 Mortar (Series 158).

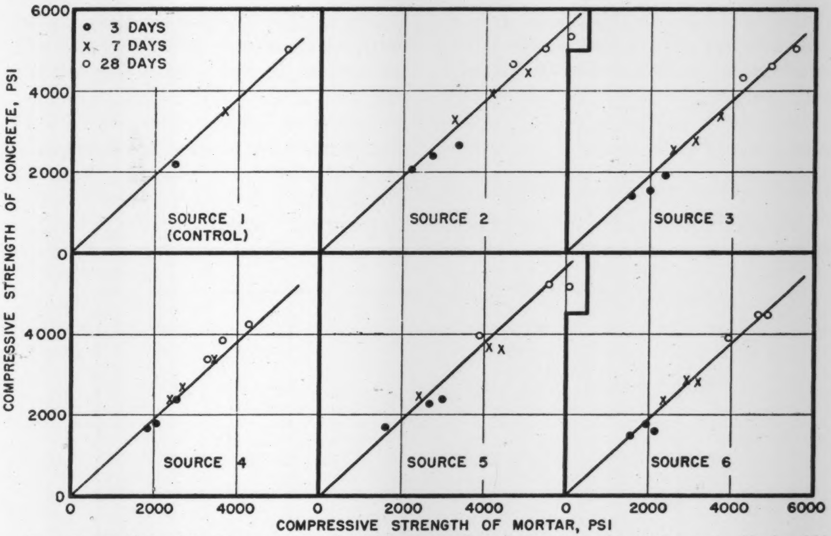

FIG. 6.—Relationship Between Compressive Strengths of Constant-Slump Concrete (Series 158 C) and C 109 Mortar (Series 158) for Individual Cement Sources.

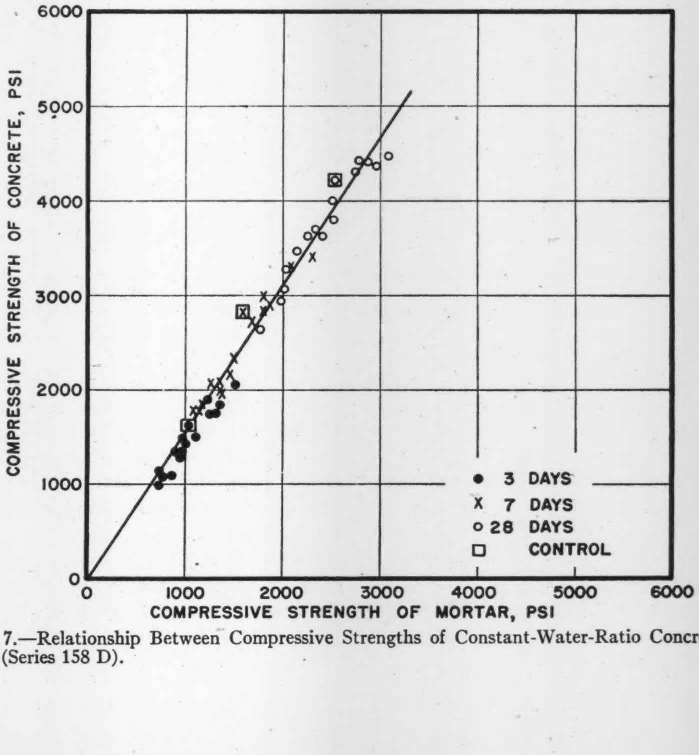

FIG. 7.—Relationship Between Compressive Strengths of Constant-Water-Ratio Concrete and Mortar (Series 158 D).

at the three ages. In the case of the control cement, the range in strength was so small at all ages that no relationship is indicated, suggesting that its strength differences were due to minor testing variations.

Strength of Concrete Versus Mortar:

Comparisons of concrete and mortar strengths are shown in Tables III and IV (*a*) and (*b*) and in Figs. 5, 6, and 7. A remarkably good correlation is shown between the two measures—concrete and mortar—of the strength-producing properties of the cements.

Table III and Figs. 5 and 6 compare the strengths of the $5\frac{1}{2}$-sack constant-slump concrete made in January and February of 1957 with the ASTM C 109 mortar made during the period from October, 1955 to October, 1956. As already pointed out, only three samples for each of the five sources were used in this concrete—representing the high, median, and low strengths revealed by the mortar tests. Figure 5, where points for all sources are plotted, shows an excellent relationship between concrete and mortar strengths, and Fig. 6 shows a similarly good one for each source individually. While a large proportion of the spread in strengths is due to the inclusion of the three test ages, it is apparent from Figs. 5 and 6 that the mortar provided a good indication of concrete strength at each age.

Table IV and Fig. 7 compare the constant-water-ratio concretes and mortars. Again three samples from each of the five sources were tested, selected with

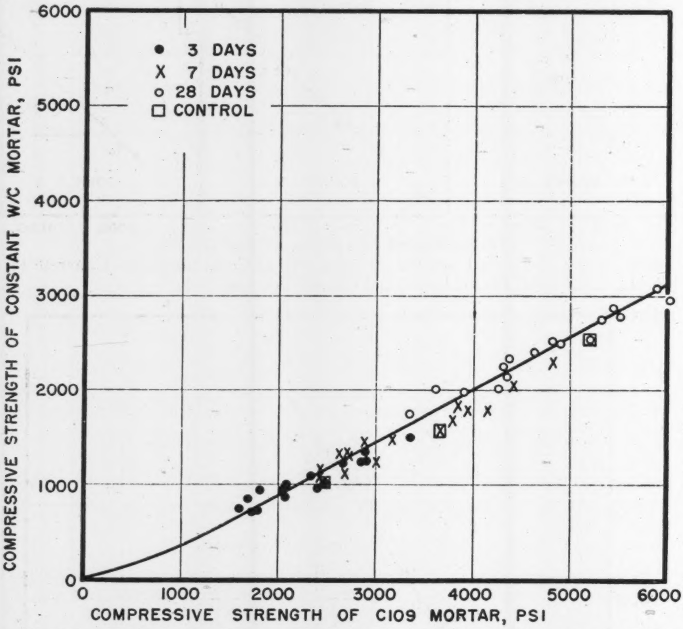

FIG. 8.—Relationship Between Compressive Strengths of Constant Water-Cement Ratio Mortar (Series 158D) and C 109 Mortar (Series 158).

principal emphasis on water demand. The slumps of concrete and the flows of mortar were allowed to vary as influenced by the water requirements of the cement. The correlation is even better than shown in Figs. 5 and 6. The diagram for the individual cements, corresponding to Fig. 6, has not been included, but the line in Fig. 7 fits the individual cements excellently.

The constant-water-ratio concrete in Table IV can be compared with the ASTM C 109 mortar for the appropriate samples from Table I. Diagrams making such comparisons are not included in this paper, but they show a strength correlation substantially as good as for Figs. 5, 6, and 7—perhaps somewhat better than Fig. 5 and not quite as good as Fig. 7. That this should be so is made clear by Fig. 8 where the strengths of the constant-water-ratio mortar are shown in relation to those for the C 109 mortar.

TABLE V.—EXPECTED VARIATIONS IN STRENGTH OF CEMENTS FROM DIFFERENT SOURCES.

Values based on normal probability distribution using 28-day sample-to-sample standard deviations shown in Table I.

Cement	Deviation Below Average Strength, to be Expected					
	1 time in 10		1 time in 20		1 time in 100	
	psi	per cent	psi	per cent	psi	per cent
Source 1[a]	158	3.1	202	3.9	286	5.6
Source 2	435	8.1	558	10.4	789	14.7
Source 3	464	9.4	595	12.0	842	17.0
Source 4	313	8.5	401	10.9	568	15.4
Source 5	667	12.3	855	15.8	1210	22.3
Source 6	314	6.8	403	8.7	570	12.3
Average 2 to 6	439	9.0	562	11.6	796	16.3

[a] Control sample, blend of 5 brands.

Significance of Variations:

The significance of variations in cement from the same mill is well illustrated in Table III. For the 28-day tests, the average strengths of the five cements from outside sources ranged from a high of 4880 to a low of 4060, an over-all spread of 820 psi. It is of interest from a practical standpoint to compare this variation with those due to other factors.

The difference between 4880 psi and 4060 psi can mean a difference in required cement content of about $\frac{3}{4}$ of a sack per cu yd. That is to say, the cement requirement would have to be increased by this amount if the needed average strength were 4880 psi, but the cements were so variable as to drop periodically to the lower level of strength-producing potential. From the age-strength data in Table III, a comparison can be made between the times required for high- and low-testing cements to attain a given strength. The lower testing samples from the various sources required 28 days to attain an average strength of 4060 psi, whereas the high-testing shipments could be expected, from the relationship shown, to provide this same strength in only about 12 days.

The variability in strength contributed by within-source cement variations can also be compared with that resulting from poor control of mixing water. If concrete intended to have a slump of 1 to 2 in. were allowed to be used with 6 to 7 in., the resulting strength reduction would be about the same as the difference shown in this investigation between average high- and low-testing samples from the same source. Recent data on strength-producing properties of coarse aggregates provide another basis for evaluating the contribution of cement variability to strength variations.[3] Sixteen coarse aggregates, differing widely in origin and physical characteristics, produced compressive strength levels ranging from 8 per cent below to 8 per cent above average in concretes of the same cement factor and slump. This range for completely different aggregates is smaller than that shown here for different shipments of cement from the same source.

Still another approach to evaluating the variability of the different cement sources is illustrated in Table V. It shows the amount by which the strength for individual samples may be expected to fall below the average strength for the particular source one time in 10, one in 20, and one in 100. For example, the data show that only one time in 100 would the 28-day mortar strength for the "control" cement be expected to fall below the average by as much as 286 psi or 5.6 per cent. For the outside sources, the minimum strength deficiency for the one in 100 chance was about 570 psi or 12.3 per cent, and the maximum was

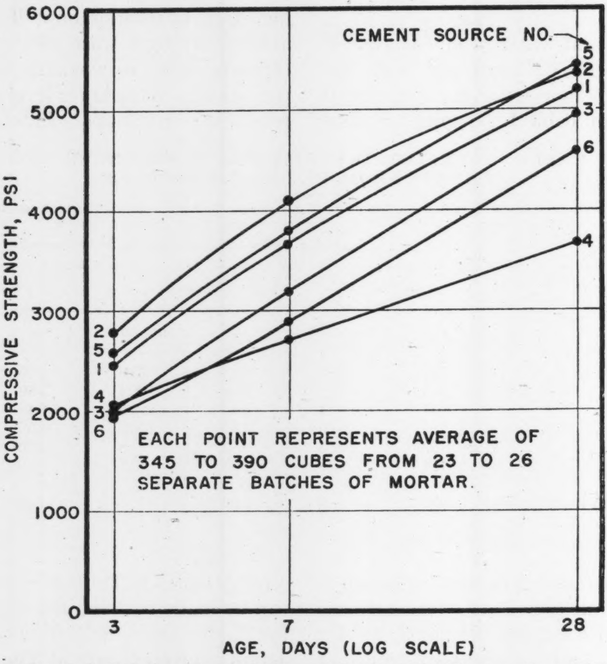

Fig. 9.—Age-Strength Relationships for Mortars Made with Cements from Different Sources (Series 158).

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[3] "Studies of Flexural Strength of Concrete, Part I—Effects of Different Gravels and Cements," *Joint Research Laboratory Publication No. 1*, The National Sand and Gravel Assn., and the National Ready Mixed Concrete Assn.

1210 psi or 22.3 per cent for cements 6 and 5, respectively.

Although concrete strength variations are caused by a variety of factors, it is clear that lack of uniformity of cement ranks high among them.

Effect of Age on Strength and Strength Variation:

Age-strength relationships for the six cement sources are shown in Fig. 9. The slopes of all the curves are substantially the same except for cement No. 4, which displayed slower strength development at later ages than the others. The curves also demonstrate the wide range in strength levels attained by the different cements, with the spread amounting to about 800 psi at 3 days, 1300 psi at 7 days, and 1700 psi at 28 days.

TABLE VI.—EFFECT OF VARIATIONS IN MIXING WATER ON FLOW AND STRENGTH OF ASTM METHOD C 109 MORTAR (SERIES D55).

Tests made in strict accordance with ASTM Method C 109 except that quantity of mixing water was fixed at various levels to determine effect on flow and strength.

Batches consisted of 740 g of cement (Lot 3081, blend of 5 brands used for this series only), 2035 g of graded standard sand and indicated quantity of mixing water. Each value average for 3 batches mixed on different days; compressive strengths are averages for 9 2-in. cubes, 3 from each batch tested at each age.

Mixing Water, ml	Water-Cement Ratio by Weight	Flow, per cent	Compressive Strength, psi		
			3 Days	7 Days	28 Days
330.....	0.446	80	2530	3735	5056
340.....	0.459	91	2434	3572	5035
350.....	0.473	102	2349	3460	4922
360.....	0.486	112	2243	3502	4973
370.....	0.500	120	2178	3404	4852
380.....	0.514	130	2130	3259	4834

Although, as shown in Table I, strength variations followed the same trends at all test ages, the magnitude of percentage variability became less as age increased. Sample-to-sample coefficients of variation displayed a consistent downward trend with increasing age. However, it is interesting to observe that the downward trend in coefficient of variation, particularly between 7 and 28 days, is due only to the increase in average strength, not to a reduced standard deviation.

Miscellaneous Tests:

To provide background information on the significance of variations in mixing water and consistency of mortar and concrete, as well as to check on possible sources of variation in mortar strength test results, two supplementary groups of tests were made. Data from the first group, which was intended to show the relationship of changes in mixing water of ASTM C 109 mortar to flow and strength, are shown in Table VI and Fig. 10.

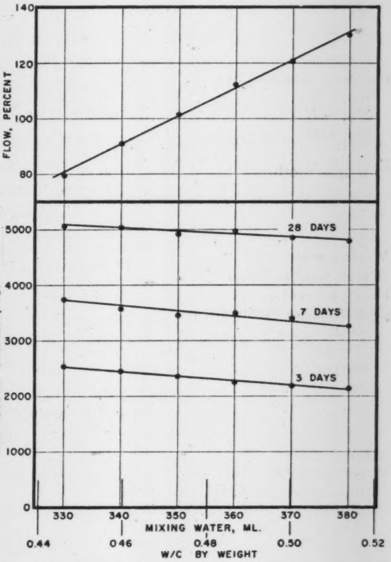

FIG. 10.—Effects of Mixing Water Variation on Flow and Strength of C 109 Mortar (Series D-55).

Figure 10 shows that the flow of ASTM C 109 mortar was changed one percentage point for each 1 ml difference in the amount of mixing water in a standard 9-cube batch. As was to be expected, increasing the flow by increasing the mixing water caused the compressive strength to be reduced. However, over the range in flow of 100 to 115 permitted by ASTM Method C 109, the effect on strength was quite small. At 28 days, the data show a reduction in strength of only about 100 psi or 2 per cent for an increase in flow from the permissible minimum of 100 to the permissible maximum of 115.

TABLE VII.—RESULTS OF MORTAR STRENGTH TESTS FOR DIFFERENT GRADINGS OF OTTAWA SAND.

Tests made in accordance with ASTM Method C 109. For each sand grading, 3 batches of mortar were made, from each of which 9 2-in. cubes were molded and tested in groups of 3 at 3, 7 and 28 days. Values are averages for the 3 batches.

Item	Sand Grading			
	Fine	Medium	Coarse	Average
Per cent retained on:				
No. 30	0	2	4	..
No. 50	60	72	80	..
No. 100	95	98	100	..
Fineness modulus	1.55	1.72	1.84	..
ACTUAL TEST DATA				
Mixing water, ml[a]	367	355	345	356
Water-cement ratio	0.496	0.480	0.466	0.481
Flow, per cent	112	107	103	107
Compressive strength, psi:				
3-day	2273	2365	2461	2366
7-day	3494	3633	3722	3616
28-day	4937	4982	5218	5046
DATA ADJUSTED TO UNIFORM FLOW OF 107[b]				
Mixing water, ml	362	355	349	355
Water-cement ratio	0.489	0.480	0.472	0.480
Compressive strength, psi:				
3-day	2314	2365	2426	2368
7-day	3543	3633	3680	3619
28-day	4960	4982	5198	5047
Compressive strength, per cent of average:				
3-day	98	100	102	..
7-day	98	100	102	..
28-day	98	99	103	..
Average	98	100	102	..

[a] Mixing water used in 9-cube batch with 740 g cement and 2035 g graded standard sand.
[b] Based on data in Table VI and Fig. 10.

Table VII gives data from a study of the effect of variations in sand grading on the strength of ASTM C 109 mortar. During the course of the principal mortar strength investigation, all bags of the standard sand were tested for sieve analysis. At least three tests were made on each bag, from the top, middle, and bottom, to assure that any variations due to segregation, as well as those due to differences between bags, would be detected.

Out of the 169 sieve analyses, there were 15 cases of minor violations of the ASTM grading requirements. In 12, the percentage retained on the No. 50 sieve fell below the minimum limit of 67 per cent (least value 64.0 per cent) and twice it exceeded the maximum of 77 per cent (greatest value 78.9 per cent). One test showed only 95.4 per cent retained on the No. 100 sieve, a technical violation of the 96 per cent minimum. In spite of the variations of individual sand samples, there was only one case where the average sieve analysis for an entire bag was outside the prescribed limits. In that instance, the average percentage passing the No. 50 sieve was 65.5 per cent, whereas the permissible minimum is 67.

Since the sand was sampled dry, it is probable that much of the variation in grading could be attributed to segregation of the test samples. That effect would be expected to be much less in the larger portions of sand used in mortar. Nevertheless, to determine if there was any possibility of sand grading having been a factor in strength test results, a supplementary group of mortar tests was made.

Mortars were prepared in accordance with ASTM Method C 109 using three sand gradings produced by sieving the sand into individual sizes and recombining it for each batch. One grading was finer than the finest individual test (fineness modulus 1.55 as opposed to 1.60); one was coarser than the coarsest test (fineness modulus 1.84 as opposed to 1.82); and one represented the median of the ASTM limits (fineness modulus 1.72). Three batches were made on different days with each grading. Nine 2-in. cubes were molded from each batch for tests in triplicate at ages of 3, 7, and 28 days.

As evidenced by the water-cement ratios in Table VII, mixing water requirements of the mortars decreased as the sand became coarser. However, there was also an accompanying reduction in flow which, although within the tolerances of ASTM Method C 109, accounted for some of the difference in mixing water and, hence, in strength. Therefore, in the lower portion of the table, water ratios and strengths have been adjusted to a flow of 107 on the basis of the data presented in Table VI and Fig. 10.

The net effects of grading differences on strength were relatively small. Based on averages for the three test ages, corrected for differences in flow, strengths ranged only from 2 per cent lower to 2 per cent higher than the average. Without correction, the range was only about ±3 per cent. When it is considered that these data encompass more than the extreme range in grading of individual sand samples, it is clear that variations in strength displayed by the different cements were not attributable in any significant degree to variability of sand grading. This conclusion is reinforced by the fact, discussed earlier, that all strength variations for the "control" cement were very small, as were also the differences from batch to batch for individual samples of a given cement.

Variations in Mixing Water Requirements:

ASTM Method C 109 requires that flow of the mortar be maintained between 100 and 115, with mixing water being adjusted to permit staying within those limits. Table II gives the quantities of mixing water used in the original mortar tests and the resulting flows for the standard 9-cube batches. Values are averages of the five batches mixed with each sample.

Since the flow table was calibrated periodically and testing conditions and temperatures were very closely controlled, it would appear that the required mixing water for the uniform "control" cement should have remained constant.

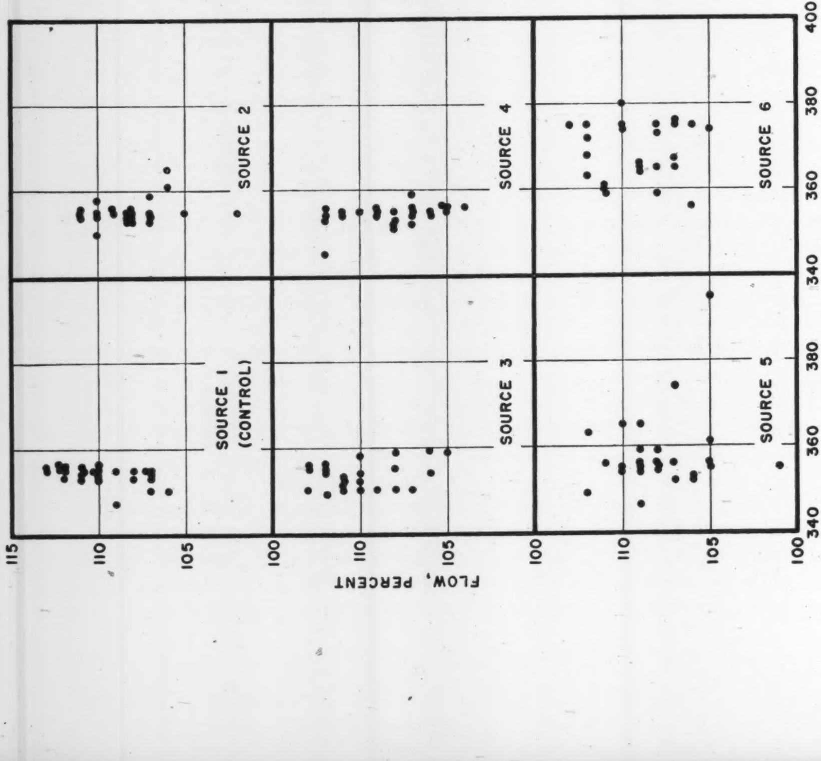

FIG. 11.—Relationships of Mixing Water to Flow of Mortar (Series 158).

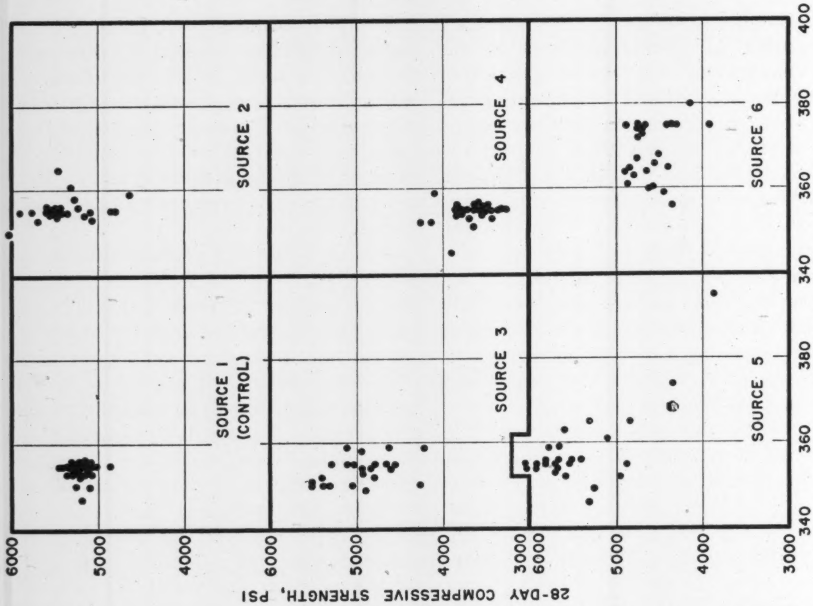

FIG. 12.—Relationships of Mixing Water to Strength of Mortar (Series 158).

While there were minor variations, probably due to personal equation and, perhaps, also differences in humidity, the average mixing water for the 5 batches representing a given sampling period rarely deviated more than 1 or 2 ml from the over-all average of 354.

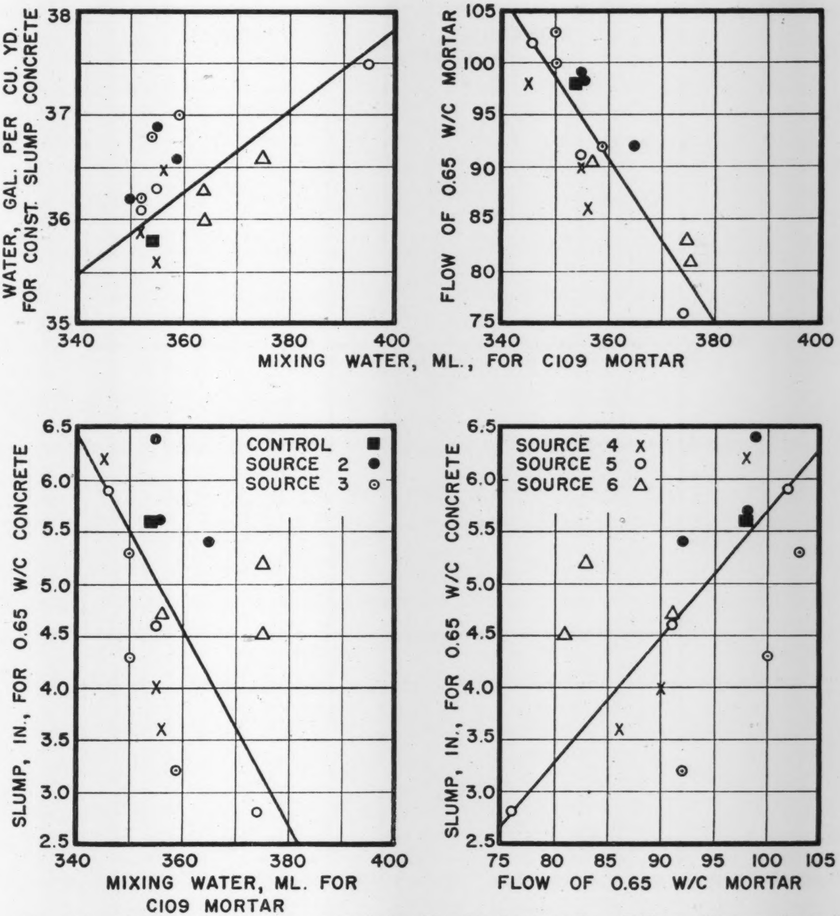

FIG. 13.—Mixing Water-Consistency Relationships for Cements from Different Sources (Series 158, 158C, and 158D).

All outside sources showed a larger spread in mixing water than the "control," although perhaps not significantly so for cement No. 2 (range 15 ml), No. 3 (10 ml) and No. 4 (14 ml). The ranges for cements 5 and 6 appeared to be significantly greater, being 49 and 24 ml, respectively. However, in the case of cement No. 5, the range is reduced to only 16 ml if the last two samples are omitted. The different variations in water demand were of necessity accompanied by about the same variations in flow for all cements because of the flow limitations imposed by ASTM Method C 109.

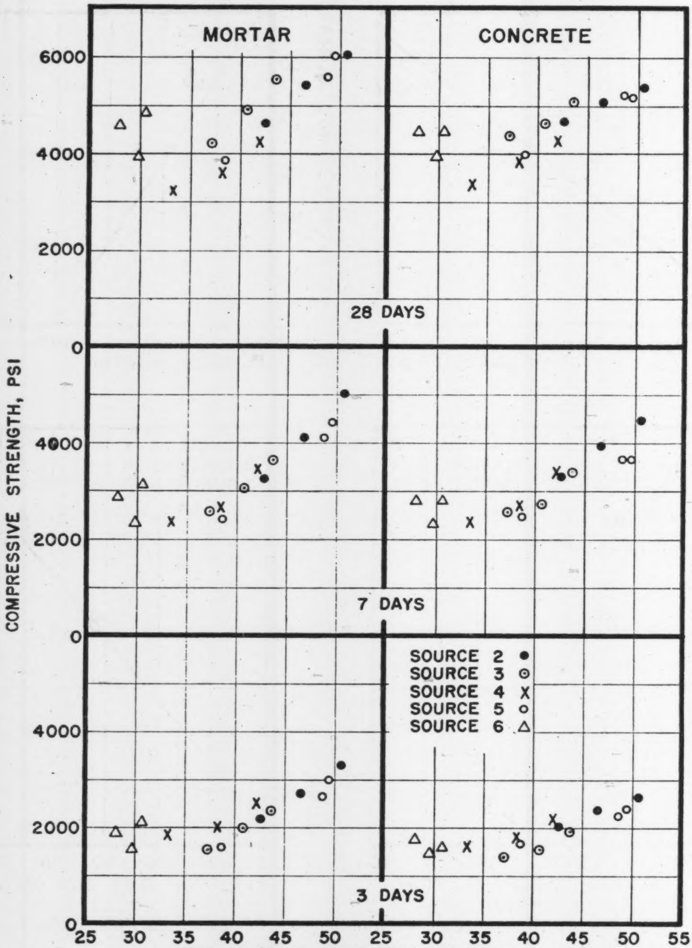

FIG. 14.—Relationship Between Strength and Tricalcium Silicate Content of Cements (Series 158 and 158 C).

Figure 11, in which flow is plotted against mixing water content, appears to show that, within the range permitted by ASTM Method C 109, the variations in flow were not related to the changes in quantity of mixing water. The grouping of points in narrow vertical bands for cements 1, 2, 3, and 4 suggests that, for them, a fixed quantity of mixing water probably could have been used without significantly increasing the spread in flow. For cements 5 and 6, however, it appears that the changes in mixing water were necessary to maintain the flow within the prescribed limits. In other words, Fig. 11 bears out the inference made above that only cements 5 and 6 showed evidence of having important variations in mixing water demand from shipment to shipment.

It might be thought that changes in water-cement ratio of mortar resulting from variations in water demand could account for some or all of the sample-to-sample strength variations of cement. However, as shown earlier, in Fig. 10, strength was only slightly affected by rather large intentional differences in water and flow. Further evidence that strengths were not generally related to water demand is provided in Fig. 12 where strength of ASTM C 109 mortar is plotted in relation to mixing water content. Only in the case of source No. 5 is there evidence that the large variations in strength, found for all cements, were accompanied by changes in water demand. For that cement, the two samples which required significantly more than average mixing water also produced the lowest strengths. With these two samples included, a reasonably convincing curve could be drawn through the points showing an inverse relationship of strength to water requirement; without them, no relationship exists.

Miscellaneous Relationships:

Some miscellaneous relationships are shown in Figs. 13 and 14. In the upper left corner of Fig. 13, the mixing water requirements of ASTM C 109 mortar are plotted in relation to the mixing water requirements of the constant-slump concrete. Were it not for the erratic sample No. 26 from source 5, no relationship would be discernible, and the one shown is of doubtful validity.

The rather good relationship in the upper right corner between water requirement of ASTM C 109 mortar and flow of constant-water ratio mortar suggests that there were real differences in mixing water requirement of mortar from shipment to shipment. At the same time, it appears from the other portions of the diagram that these differences are reflected to only a limited degree in concrete. This should probably be expected since the effects of cement differences on consistency would logically be less pronounced in concrete than in mortar.

Nevertheless, it is interesting to observe that, while no good over-all correlation exists between mortar and concrete so far as consistency or water demand is concerned, the cements from individual sources do tend to reveal a consistent pattern. For example, for all sources but No. 6, the slump of constant-water-ratio concrete decreased progressively with increasing water requirement of the ASTM C 109 mortar as shown in the lower left portion of Fig. 13. Similarly, for individual cement sources, the slump of constant-water-ratio concrete tended to increase as the flow of the constant-water-ratio mortar increased, as shown in lower right.

In considering the significance of strength comparisons based on the two types of concrete (fixed water ratio or constant slump), it is of interest to note that cements which produced low strengths also tended to have low slumps in the fixed water ratio concrete. Thus, if the water had been increased to provide a constant slump, as would be necessary under field conditions, strength deviations from the median would have been even greater than indicated.

TABLE VIII.—CHEMICAL ANALYSES, COMPOUND COMPOSITIONS, AND FINENESS TESTS OF CEMENTS (SERIES 158 AND 158 C).

Oxide analyses made by Research Laboratory of the Portland Cement Assn. Fineness values averages for tests by the Portland Cement Assn. and the National Ready Mixed Concrete Assn., using air permeability, ASTM Method C 204.[c]

Cement	Sample	Strength Level[a]	Chemical Analysis, per cent												Calculated Compound Composition, per cent[b]				Fineness, sq cm per g
			SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	SO_3	Na_2O	K_2O	Loss on Ignition	Insoluble Residue	Mn_2O_3	Free CaO	C_3S	C_2S	C_3A	C_4AF	
Source 2......	No. 15	High	20.47	6.17	2.23	63.79	2.85	2.17	0.07	0.84	1.45	0.15	0.05	0.61	50.7	20.5	12.6	6.8	3400
	No. 8	Median	20.67	5.96	2.35	63.62	2.74	2.12	0.12	1.02	1.57	0.25	0.04	1.43	46.6	24.2	11.8	7.1	3355
	No. 5	Low	21.27	5.45	2.60	62.54	2.43	2.24	0.12	0.90	2.54	0.43	0.09	0.88	42.6	28.9	10.1	7.9	3460
Source 3......	No. 11	High	21.22	5.89	1.88	63.54	2.70	2.00	0.08	0.14	1.70	0.22	0.67	1.40	43.7	28.0	12.4	5.7	3695
	No. 9	Median	21.44	6.08	1.88	63.47	2.55	2.02	0.09	0.15	1.66	0.22	0.57	1.33	40.7	30.8	12.9	5.7	3600
	No. 26	Low	22.03	5.90	1.86	63.45	2.64	1.74	0.10	0.15	1.34	0.27	0.66	1.56	37.2	35.2	12.5	5.7	3510
Source 4......	No. 15	High	21.23	4.96	2.13	62.25	4.85	1.74	0.53	0.96	1.25	0.35	0.04	2.14	42.0	29.3	9.5	6.5	3590
	No. 22	Median	21.84	4.29	3.10	61.43	4.50	2.41	0.44	0.90	1.12	0.29	0.02	1.37	38.3	33.8	6.1	9.4	3350
	No. 14	Low	21.10	5.25	3.15	60.22	4.54	2.57	0.57	1.08	1.63	0.37	0.03	1.07	33.3	35.5	8.6	9.6	3290
Source 5......	No. 2	High	21.12	6.17	2.14	65.16	1.36	2.28	0.08	0.54	1.00	0.07	0.18	1.00	49.6	23.2	12.7	6.5	3110
	No. 15	Median	21.24	6.13	2.20	65.32	1.36	1.99	0.08	0.68	0.86	0.06	0.19	1.38	48.8	24.2	12.5	6.7	3230
	No. 26	Low	21.47	6.33	2.27	64.02	1.10	2.41	0.09	0.55	1.62	0.40	0.14	1.51	38.6	32.5	12.9	6.9	3270
Source 6......	No. 2	High	21.74	6.36	2.86	62.54	2.07	2.44	0.27	0.71	0.79	0.14	0.04	1.24	30.5	39.4	12.0	8.7	3645
	No. 18	Median	21.99	6.21	2.59	62.20	1.93	2.55	0.25	0.64	1.08	0.30	0.06	1.33	28.0	42.0	12.1	7.9	4075
	No. 14	Low	21.71	6.32	2.76	62.20	2.02	2.65	0.26	0.68	0.77	0.17	0.04	1.10	29.7	40.0	12.1	8.4	3420

[a] High, median, and low testing of samples taken at approximately 2-week intervals from each source, based on strength level of ASTM C 109 mortar (see Table 1).

[b] Corrected for free lime.

[c] 1955 Book of ASTM Standards, Part 3, p. 145.

Relationship of Cement Strength to Compound Composition:

Table VIII shows oxide analyses made by the laboratory of the Portland Cement Assn., compound compositions calculated therefrom, and results of fineness tests for the three cement samples from each outside source which had produced highest, median, and lowest strengths in the ASTM C 109 mortar. In Fig. 14, the mortar and concrete strengths for these samples have been plotted in relation to tricalcium silicate content. Except for cement source No. 6, there is a good general relationship showing increased strength with increased percentages of tricalcium silicate. In fact, for sources 2 through 5, the mortar strengths at each test age fall in exactly the same order as the percentages of tricalcium silicate. The same is true for concrete strengths except in the case of source 5 where the difference between the high and median tricalcium silicate content was less than one percentage point and the corresponding concrete strengths were essentially the same. For source No. 6, the over-all range in tricalcium silicate was quite small and the spread in fineness was greater than for any other source. These factors may account for the apparent lack of a relationship between tricalcium silicate and strength for that cement.

The chemical analyses strongly suggest that there were variations in the manufacture of the cements which were reflected in their chemical composition and which caused different shipments to have different strength-producing properties.

CONCLUSION

Data from the several groups of tests, involving mortars and concretes made with a large number of cement samples from several sources, show the following:

1. Shipment-to-shipment variations in strength-producing properties of cements from five commercial sources were large in relation to the variations which could be attributed to testing differences.
2. Differences in strength, as measured by tests of standard mortar, were reflected in the strength of concrete.
3. The variability in strength for all five cement sources was sufficiently great to be of considerable importance in controlling the strength of concrete.
4. The pattern of strength variations for a given source of cement was similar at all test ages. That is, samples which were low-testing at one age were also low at the other ages.
5. The well-known differences in strength development of cements from different sources were again demonstrated here. Based on the average of all samples and all test ages, there was a spread, between the highest and lowest testing sources, of 45 per cent of the over-all average mortar strength.
6. Sample-to-sample standard deviations (psi) tended to remain fairly constant at all test ages for a given source of cement. Because of the increase in strength with age, there were progressive reductions in coefficients of variation (in per cent) as age increased.
7. Except for source No. 5, the shipment-to-shipment variations in water demand for mortars were small and their reflection in the slump of concrete was probably of little practical significance. In any event, the importance of consistency variations were greatly overshadowed by the large variations in strength.
8. Supplementary tests showed conclusively that normal variations in consistency, within the limits permitted by ASTM Method C 109, or variations in grading of standard sand during the progress of the work, could not have

been a significant source of variations in mortar strength.

9. There was strong evidence that differences in strength-producing properties of cements were directly related to their tricalcium silicate content. It may be, therefore, that chemical analyses could be used to provide advance warning of probable strength variations.

10. This research points to the need for better control of the uniformity of cement production within individual sources.

Acknowledgments:

The investigations were outlined and carried out under the direction of the authors in accordance with the scope established by the Association's Committee on Cement. That committee consists of R. C. Collins (chairman), Fred F. Bartel, Herbert Jahncke, J. A. Nicholson, E. J. Nunan, F. E. Schouweiler, M. Eugene Sundt, and Stanton Walker.

The first portion of the tests was conducted under the supervision of James F. Shook, then Associate Research Engineer in charge of laboratory and now Materials Engineer, Am. Assn. of State Highway Officials Road Test, Ottawa, Ill. The latter portion of the tests was conducted under the supervision of Richard D. Gaynor, Associate Research Engineer who succeeded Mr. Shook as Laboratory Manager. All mortar specimens were prepared by Herman L. Knoppel, Jr., Assistant Laboratory Supervisor, a highly skilled technician specially trained for this particular work.

DISCUSSION

MR. R. H. BRINK[1] (*presented in written form*).—The paper provides data that have been needed for some time. Those concerned with the production or use of concrete soon recognize that some variations in the strength of concrete are not related to such factors as aggregates, mix design, or the handling process. The real cause of such variations often goes unidentified because we are prone to suspect improper handling or testing of test specimens whenever no other explanation is apparent. The authors are to be congratulated for the effective manner in which they have defined that part of the observed variations which should be attributed to nonuniformity of the cements themselves as opposed to that inherent in the testing procedure.

The interest of the Bureau of Public Roads in this subject has been heightened in recent years by the concern shown by some state highway departments over the variable behavior of cement from the same mill. In a survey covering 43 states conducted in 1955, it was found that 13 state highway departments made extensive use of type II instead of type I cement, and 7 states had introduced modifications of the usual requirements for type I or II cement. A need for greater uniformity in finishing characteristics and strength development generally accounted for the reluctance of these states to use normal type I cement.

A question arises as to the feasibility of attempting to control the variability of cement by such measurable factors as chemical composition and fineness. To study this matter, test results, originally obtained through the normal acceptance testing program of a state highway laboratory, were examined. The variability of the 21 different cements represented is of particular interest because the governing specifications limited the tricalcium silicate to 50 per cent, the tricalcium aluminate to 15 per cent, and the turbidimeter fineness to 2100 sq cm per g.

The accompanying Fig. 15 shows the coefficient of variation of the 28-day compressive strength of mortar for all samples meeting the above requirements. Each bar on the lower axis represents all samples of cement obtained from a single mill over a period of several years. The testing variation indicated is that found by Walker and Bloem to be the sample-to-sample variation for their control cement. This factor would vary slightly from laboratory to laboratory. It is evident that most of the cements represented show considerable variation above that which can be attributed to testing variation even though they were subject to the additional requirements mentioned above.

One of the conclusions of the paper under discussion points to a relation between strength and tricalcium silicate. Such a relation would seem to indicate that the uniformity of cement would be improved if close control were maintained over the tricalcium silicate content. This possibility was explored by selecting only those samples from each cement which had a tricalcium silicate content within ±2.5 per cent of the average. This procedure resulted generally in reducing the number of samples nearly 50 per cent. The differences between the coefficients of variation for this selected group of samples and the coefficients for all samples are plotted on the middle axis of Fig. 15. For 14 out of the 21 cements, the indicated differences are not considered significant because they are less than the standard error which was calculated for each coefficient of variation. Those differences which are considered significant are marked by a check and indicate that the variability of some, but not all, cements can be lessened by closer control of the tricalcium silicate content.

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[1] Physical Research Engineer, Bureau of Public Roads, Washington, D. C.

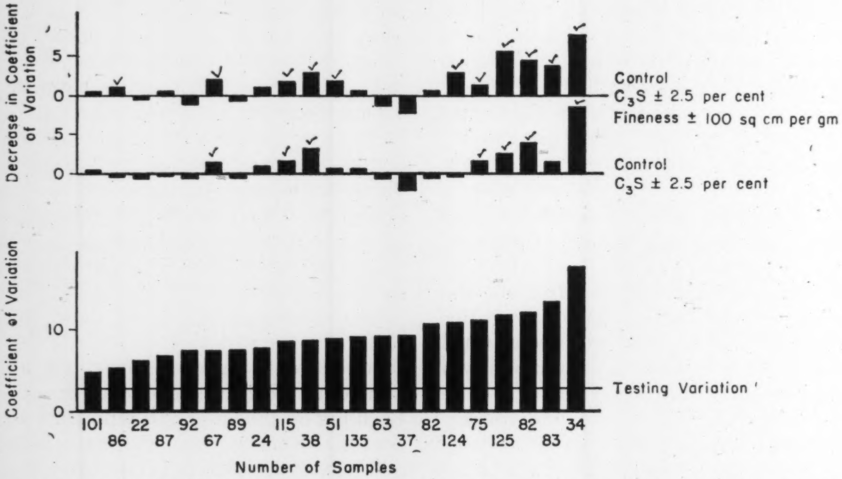

FIG. 15.—Variation of 28-day Compressive Strength of Mortar for Cement from the Same Mill.

The additional effect of controlling fineness more closely was also investigated. From the samples selected for their uniform tricalcium silicate content, a further refinement was made by selecting only those samples which did not vary in fineness more than ±100 sq cm per g from the average. The differences between these coefficients and those for all samples are plotted on the top axis of Fig. 15. With both the tricalcium silicate and fineness closely controlled, significant decreases in variation were found for 11 of the 21 cements. For only 5 cements, however, was the additional decrease due to control of fineness as much as 1 per cent over that due to control of C_3S content alone. This analysis indicates that a large part of the variation in strength-producing properties of cement results from variations in the manufacturing process itself.

Slightly less variation would probably have been indicated for each cement if the number of samples collected during each year had been large enough to permit basing the coefficients on single rather than several years' production. Some idea of the magnitude of this reduction can be obtained by the comparison afforded in Fig. 16 where the coefficients of variation of four cements are shown as calculated by the two methods. The left bar for each cement represents the variation of samples over a 3-yr period whereas the right bar is an average of the variation among samples obtained during each of the 3 yr.

The authors have amply demonstrated the difficulties that may arise when the strength of concrete is subject to variation as a result of variable cement. It should be noted that variation in finishing characteristics, or more specifically rate of hardening, may be equally important for the proper conduct of highway paving operations. Variations in the setting characteristics becomes most critical when temperatures are high. The increased use of retarding admixtures has also served to focus attention on the variable rate of hardening of cement from the same mill. Variation in setting properties is more difficult to define than variation in strength but is a matter equally deserving of study.

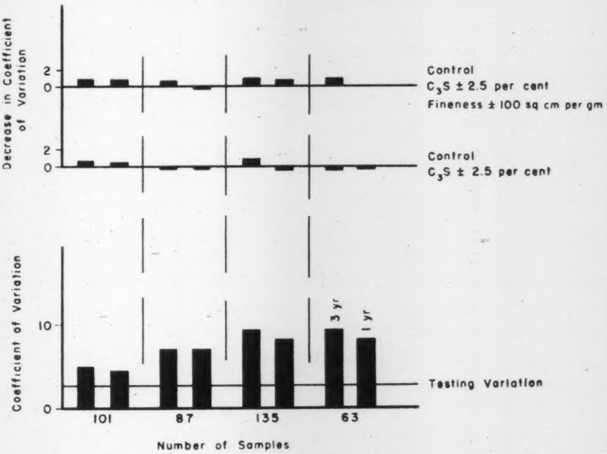

FIG. 16.—Variation of 28-day Compressive Strength of Mortar for Cement from the Same Mill

MR. M. A. SWAYZE.[2]—As the authors point out, it is a well-known fact that from brand to brand over this country you will find various inherent strengths in cements. That is natural. Raw materials from different sources in the country have very definite cement-making properties. When it is considered that the differences in composition in going from one area to another, it is surprising that we have the uniformity that is shown between brands.

Then there is potential composition. You will find certain areas in this country where all of the producers in the area have a composition of raw material which lends itself very handily to production of high inherent strengths. By this means you will often find in these areas higher strengths than in other sections of the country.

Coming down to the variation in a single plant's product, a good deal depends upon the methods of controlling quality. There are still some plants controlling on the basis of tensile strength of mortars. Nothing is more apt to give high variation in compressive strength of concrete than the assumption that because you have uniform tensile strength you are going to have a uniform concrete-making strength. There is another point that the authors brought out. The tricalcium silicate content of cement has a direct bearing on the strength-giving properties. I am surprised to hear though that variations in SO_3 content had no direct bearing on the strength properties.

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[2] Director of Research, Lone Star Cement Corp., New York, N. Y.

There is one thing necessary in the average cement plant, and that is a high degree of control of the amount of gypsum that is added to the clinker. The lower the gypsum addition, the lower the early strength development: Also the ultimate strength will be somewhat lowered.

Again, you will find some places in the industry where there are rather wide variations in the silica content of raw materials. Plants of that sort that are attempting to control their composition of raw mixes by just the analysis for the carbonate portion can have considerable variation in tricalcium silicate content. Fortunately, the day of control by calcium carbonate alone seems to be rapidly waning. With new X-ray methods of analysis of raw material mixtures, I think the day will soon come when we will design raw mixtures before they are ever burned to a constant C_3S content.

Coming back to the other part of the authors' paper, first of all I want to compliment them on their tolerance. Some of the variations they have shown must seem to some ready mix contractors to be unreasonable. On the other hand, I heartily agree with what they said, that strength alone in the design of concrete is not enough. If we could only persuade engineers and architects to design concretes on something more than strength, and low strength at that, we would be far better off in the cement industry and in the concrete industry. Thirty years ago, a 2500-lb concrete was supposed to be high-grade concrete, and it was. You could not make this concrete with less than about 6 sacks of cement per yard. There was enough dense paste present to make the concrete practically impermeable. It was more resistent to weathering conditions. Now, what is the case? Architects and engineers are still specifying 2500-lb concrete, and where it used to be good concrete, now it is poor concrete. You can make 2500-lb concrete today with 4 sacks of type I or type II cement, or you can get it with 3½ sacks of type III cement, and either one is definitely lousy concrete, if it must withstand weathering. Sometime and soon I hope we can get a meeting of minds on this question. If engineers, architects, concrete designers will either step up their strength requirements, or else put in a minimum cement factor which will allow production of impermeable concrete, I think we will be far better off. This work of the authors has put us a long way on the road to understanding in the cement and concrete industries of the needs of the situation.

MR. EDWARD A. ABDUN-NUR[3] (*by letter*).—The authors have done an outstanding piece of work in setting forth quantitatively, the variations in cement that most concrete engineers have recognized qualitatively. They are to be highly commended for this excellent piece of work.

The data indicate that there exist variations in cement from the same source, which can be detected by routine physical and chemical tests. One must agree with the authors' conclusion that there is need for better control of uniformity of cement from individual sources.

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[3] Consulting Engineer, Denver, Colo.

The control cement, which was taken all at one time, blended and stored in sealed containers, shows some variations (series 158) in spite of extreme care in handling and preparation. These variations must be attributed to personal equations of the laboratory technicians, variations in handling, variations in testing which cannot be entirely avoided with the best care, and variations due to the preparation of specimens at various time intervals. Similar unavoidable variations in the handling and testing must be allowed for the other cements, which would then reduce their apparent overall variations.

That cement could be made more uniformly is indicated by the lower variability of three of the five cements tested. The large variability exists because of the range permitted by standard specifications. If the amount of variation reported is undesirable, then narrowing the range in the specifications would force a reduction in the variability, or a limit on variability may be introduced instead.

Although the data presented in Figs. 5, 6, and 8 provide general trends, it does not seem to the writer that such trends are sufficiently reliable for definite conclusions regarding the relationships of mortar and concrete specimens, inasmuch as the mortar specimens and the concrete specimens were not prepared at the same time and were thus subject to different testing variables. Figure 7 on the other hand, which shows results of mortar and concrete specimens made concurrently, indicates a valid general relationship—the stronger mortars made stronger concretes. It would be interesting to know how the coefficients of variations of the mortars and the corresponding concretes in this series (158 D) compare.

It is true that more variables enter into concrete than into mortar, but some of these variables may counteract one anothers' effects rather than be cumulative—if not always, at least part of the time. Thus, variations in cements may greatly influence variations in concrete strengths, provided other variables do not counteract them. Because of this possibility, the writer does not understand the emphasis placed on the probabilities shown in Table V, particularly that they apply to mortars only, when most engineers are concerned more with concrete.

The authors contend that variations in aggregates do not result in as large variation in concrete strengths as do those from cements. This may be so under laboratory research conditions, but the writer has repeatedly observed in the field, variations in the plastic concrete that were large enough to be detected visually, and that were obviously due to variations in aggregates. On the other hand, variations in cement are rarely large enough to be detected visually.

The authors have provided some very thought-provoking information. It is natural that the more variable the ingredients that enter concrete, the more difficult it is to control the final product. Therefore, more uniformity is needed not only in cement but also in aggregates and other ingredients. Most frequently in actual operations, the handling of materials, batching, and mixing may introduce variables that outweigh the variables in the materials. Therefore, proper control of these operations will help to minimize the influence of the variations in the materials on the final strength.

MR. WALTER K. WAGNER[4] (*by letter*).—This investigation does a fine job of evaluating the inherent variations in cement quality from the standpoint of strength and consistency when considered over a period of time.

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[4] Engineer, Albuquerque Gravel Products Co., Albuquerque, N. Mex.

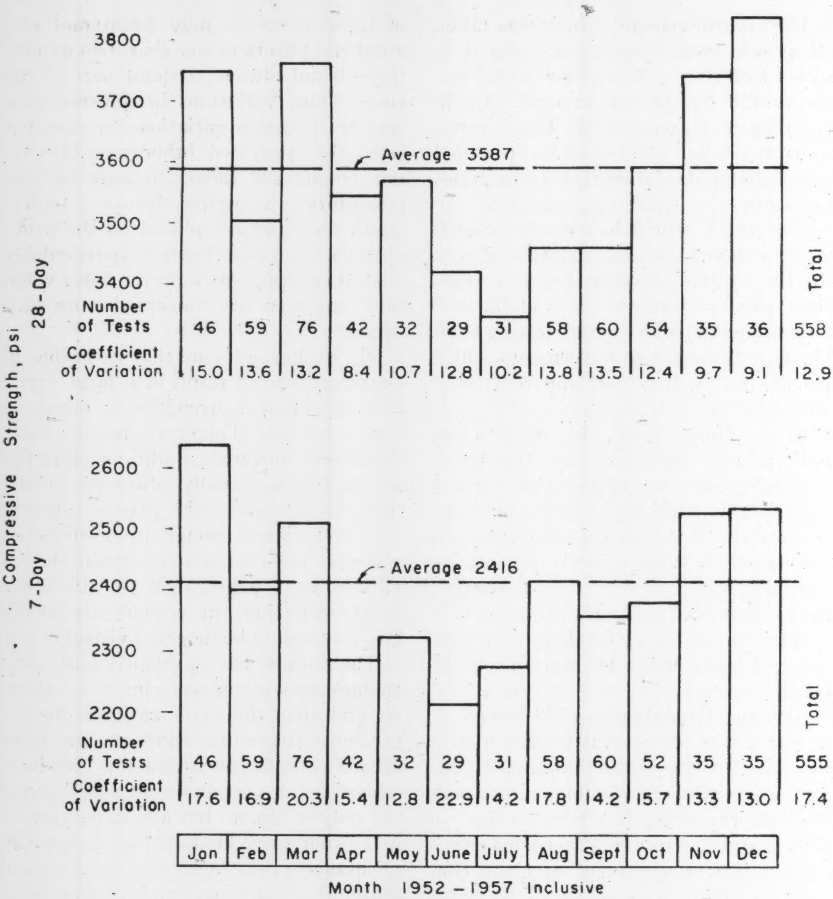

FIG. 17.—Monthly Summary of Tests by Albuquerque Testing Laboratory on AGP Commercial 3000 psi - ¾ -in. Aggregate Mix, 1952–1957 Inclusive.

ASTM Committee C-1 reported in 1955[5] on an "Investigation of the Level of Strength Development of Domestic Portland Cement." Although this investigation was made primarily to compare machine and hand-mixed mortars for ASTM Method C 109, it also showed that considerable variation existed in the quality of cements as tested by the 63 manufacturers of portland cement and 16 consumer testing agencies who participated in the program. It was stated in this report that "the Sponsoring Committee realized that the strength requirements in ASTM Specifications C 150 and C 175[6] might need revision also by reasons other than the effect of the type of mixing on the strength of mortars.

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[5] Report of ASTM Committee C-1, Appendix II, "Investigation of the Level of Strength Development of Domestic Portland Cement," *Proceedings*, Am. Soc. Testing Mats., Vol. 55, p. 378 (1955).

TABLE IX.—COMPARISON OF ACTUAL TO THEORETICAL FAILURES OF 3000 PSI ¾-IN. AGGREGATE MIX AS TESTED BY ALBUQUERQUE TESTING LABORATORY.

28-day tests. 1952–1957 inclusive.

	January	February	March	April	May	June	July	August	September	October	November	December	Total or Average
Number of tests	46	59	76	42	32	29	31	58	60	54	35	36	558
Registered strength as per cent average strength	80.0	85.6	79.8	84.7	84.0	87.8	89.7	86.8	86.8	83.7	80.3	78.3	83.5
NUMBER OF FAILURES													
1952	1	0	3	0	0	1	0	0	0	1	0	0	6
1953	1	2	2	0	0	0	0	0	0	0	0	0	5
1954	0	0	0	2	0	1	0	3	6	3	0	0	15
1955	0	3	3	0	0	0	0	0	2	1	0	0	9
1956	0	0	0	0	0	1	1	0	2	0	1	0	5
1957	1	1	0	0	0	1	4	6	0	0	0	0	13
Totals	3	6	8	2	0	4	5	9	10	5	1	0	53
PER CENT FAILURES													
Actual	6.5	10.2	10.5	4.8	0	13.8	16.1	15.5	16.7	9.3	2.9	0	9.5
Theoretical	9.1	10.4	6.6	3.4	6.4	19.5	15.5	10.6	10.6	9.0	2.3	1.0	9.6

TABLE X.—STATISTICAL DATA FOR 3000 PSI ¾-IN. AGGREGATE MIX.

Period	Age of Test	Average Strength for period, psi	Number of Tests	Standard Deviation	Coefficient of Variation
Oct., Nov., Dec., Jan., Feb., Mar., Apr.	7-day	2453	377	421	17.1
	28-day	3660	380	459	12.5
Jun., July, Aug., Sept.	7-day	2338	178	413	17.7
	28-day	3430	178	448	13.1

The Working Committee on Strength was therefore requested to make an exhaustive study of the level of strengths developed currently by domestic portland cements." It is the opinion of this writer that Committee C-1 should consider the data presented by the authors and all other available data very carefully with a purpose pointed toward an ultimate revision of cement specifications to prevent such widespread variation.

It might be argued by some persons in the industry that the situation is not serious. But this writer has recognized for a period of at least ten years that cement variation does play an important part in the control of ready-mixed concrete and last year presented a report[7] which covered several years of laboratory and field test data showing a considerable variation in car-to-car shipments of each of two brands shipped into the New Mexico market.

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[6] Specification for Portland Cement (C150 – 56), 1958 Book of ASTM Standards, Part 4. Specification for Air-Entraining Portland Cement (C 175 – 56), 1958 Book of ASTM Standards, Part 4.

[7] Walter K. Wagner, "Cement Testing Program of a Concrete Producer," presented at the 27th Annual Convention of the National Ready Mixed Concrete Assn., Los Angeles, Calif., Feb. 14, 1957.

The variation experienced in New Mexico has been a definite seasonal one. The accompanying Fig. 17 is a bar chart of the monthly summary of concrete tests made during the full six-year period of 1952 to 1957 inclusive. All tests were reported by a single commercial testing laboratory on concrete produced and sold as a commercial mix by Albuquerque Gravel Products Co. The mix was designed to produce an average strength of 3600 psi or 20 per cent over the required 3000 psi at 28 days. The design was based on water-cement ratio curves established from field tests by the same commercial laboratory in order to include all of the contingencies of batching, mixing, sampling, and testing. A coefficient of variation of 13.3 per cent and an assumed limit of failures of 10 in 100 required the 20 per cent overdesign.

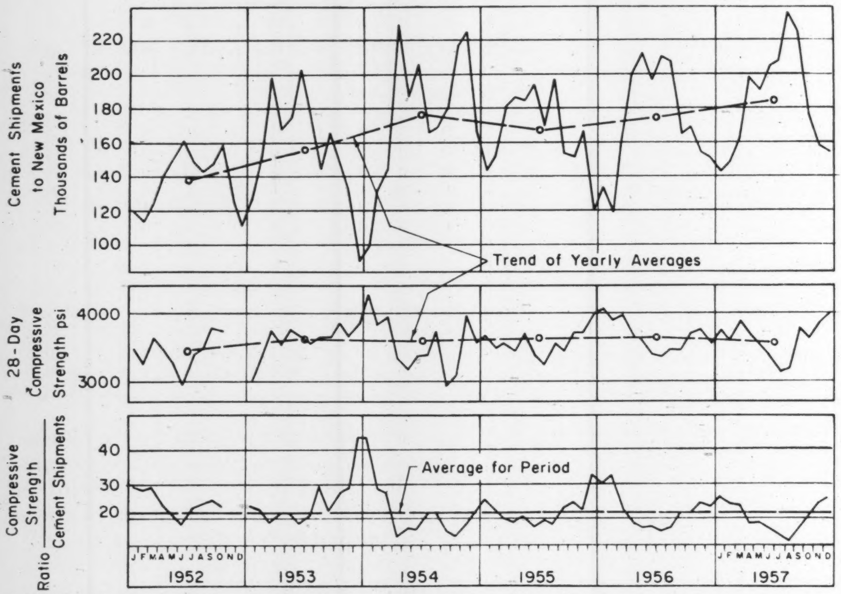

FIG. 18.—Relation of Cement Shipments to Compressive Strength of a Single Class of Concrete

A comparison of actual to theoretical failures for this period is shown in the accompanying Table IX. The close approximation of theoretical to actual failures indicates reasonably correct assumptions of our design factors. The summer months, however, or those of peak demands on the cement mills, show failures in excess of the assumed 1 in 10 while the other months generally show failures below the assumed value. If these data are analyzed for the "up quality" for the eight-month period of October–May, inclusive, and the "down quality" of the four-month period of June–September, inclusive, then approximately equal values can be obtained for coefficients of variation but a difference of 230 psi in average compressive strengths for 28 days shown in Table X.

Although this difference might not

seem significant, it amounts to almost 8 per cent of the required strength, enough to be concerned about during the summer season. This has resulted in our use of two sets of mixes for the two seasons with an average cement increase of about one third of a sack of cement per cubic yard of concrete for the summer period. At an average cost in this area of about $1.20 per sack, the added cost per cubic yard is about 40 cents. It would be difficult indeed to try to pass this cost on to the consumer. Our assumptions for design of mixes are based on practice recommended by the American Concrete Inst.[8] To use mixes throughout the year that would be adequate for the summer season only would impose an ultimate penalty on the consumer and would develop strengths of 50 per cent or more over those required during the period of maximum strength quality. Sometimes this "false security" will develop such confidence in a product by a contractor that he will take liberties in adding water because of the premium in strength. Then if quality falls off suddenly, all parties concerned can find themselves in trouble.

Figure 18 shows the relation of monthly averages of compressive strengths to shipments of cement in New Mexico during the 1952–1957 period, the latter figures having been taken from reports by the U. S. Bureau of Mines. The ratio of compressive strengths to cement shipments dramatically illustrates the seasonal variation which obviously is due to peak or slow periods of production by the mills. The comparison is shown as a direct correlation in Fig. 19. The relation might be considered quite good, especially in view of the fact that the shipments to Albuquerque Gravel Products Co. amounted to only 8.6 per cent of total cement shipments into the state for that period. It should be pointed out that there are no mills in New Mexico. Cement is shipped from California, Colorado, Kansas, Oklahoma, Texas, Utah, and Wyoming. At least 95 per cent of

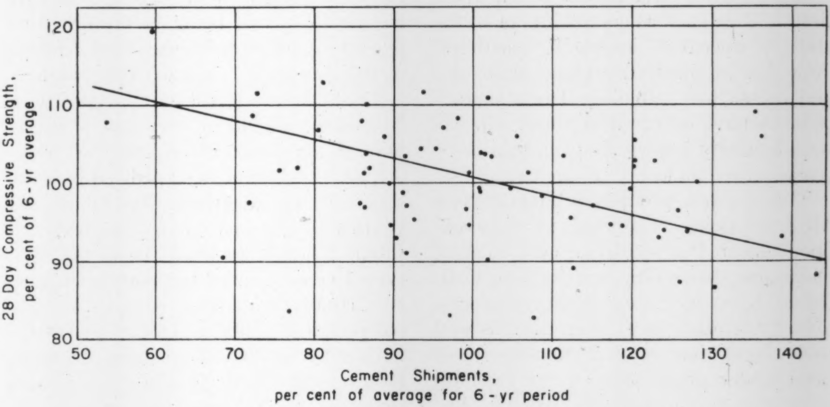

FIG. 19.—Comparison of 6-yr Averages of Cement Shipments to New Mexico and 28-day Compressive Strength of One Class of Concrete.

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[8] "Recommended Practice for Evaluation of Compression Test Results of Field Concrete (ACI 214–57)" *Proceedings*, Am. Concrete Inst., Vol. 53, p. 575 (1956–57). See also *Journal*, Am. Concrete Inst., July 1957.

the cement involved in these tests, however, was produced by two mills.

The authors' paper also brings out variations in mixing water requirements for concrete but minimizes the importance of this factor as compared to strength. This may be so for the cements involved in this investigation, but it has been the experience of this writer, both in the laboratory and in the field, that water demand has been significant from a practical standpoint.

Other factors have been apparent from time to time such as complaints of workability, uncoated aggregate particles, difficulty in finish troweling, flash set, and premature stiffening. These characteristics have occurred at times when it was definitely known that aggregates or temperature could not be the cause.

This is not a problem unique in New Mexico. Other ready-mixed concrete operators in Pennsylvania, Ohio, Texas, Oklahoma, Colorado, Arizona, and California have indicated similar performance by cements in their areas. It is well understood that cement suppliers have been under great pressure to keep up with the ever-increasing demand, but with new mills being put into service and older mills being geared for higher production, it seems that the time has come when we should expect more uniformity in the product of those mills. Committee C-1 should review present specifications with this end in mind.

MESSRS. STANTON WALKER AND DELMAR L. BLOEM (*authors' closure*).—The authors are appreciative of the constructive discussion of their paper. The unanimous agreement that there exist wider variations in the strength-producing properties of cement than are desirable augurs well for improved controls in manufacturing processes. The very nature of the raw materials from which cement is produced and of manufacturing methods makes it clear that some differences, even substantial differences, must be expected for different periods of production. On the other hand, it is equally clear that many of the differences experienced go far beyond the necessities dictated by the conditions of cement manufacture.

What can be done about it? The answer is suggested by the superior performance of two of the five mills tested. Evidently better controls existed for these two mills than for the other three. A possible answer is suggested by the new controls being incorporated in recently constructed plants and referred to briefly in Mr. Swayze's discussion.

The authors suggest that specifications for cement should be reviewed to determine if the general institution of better controls might not be expedited by revisions in specifications. We shall not venture to say just what these revisions should be, but we would point out that type I cement, used in greatest volume, is virtually unlimited as to chemical composition. This is to be contrasted with type II with its several chemical requirements. Perhaps, also, there should be some limitations on variations in characteristics: for example, an upper and lower limit on fineness and, similarly, upper and lower limits on certain of the chemical constituents. This is not an uncommon approach in many specifications.

The data presented by Mr. Brink offer most helpful evidence, supplementing that in our paper, that strength variations are associated with measurable characteristics of the cement such as fineness and amount of tricalcium silicate.

We agree wholeheartedly with Mr. Swayze regarding the over-emphasis on measured strength as the prime criterion of acceptability of concrete. Certainly, the strongest concrete is not always the best. Nevertheless, until strength is viewed in its proper perspective as only a general indication of over-all quality (and certainly not a quantitative measure of load-carrying capacity), the concrete producer must contend with the fact that arbitrary strength limits have to be met. Mr. Swayze expressed surprise that we did not find better agreement between strength and SO_3 content. That relationship was investigated but it failed to show a well-defined correlation.

Mr. Abdun-Nur's concern over the fact that the mortar and concrete tests which correlated so well were not made concurrently does not seem justified. That circumstance, if anything, would be expected to make the indicated relationship more obscure rather than to enhance it. Hence, the fact that such an excellent correlation existed, in spite of the time element, bolsters the evidence that mortar strength of cement is a direct indication of its concrete strength-making properties.

What Mr. Abdun-Nur says about the important effects of variables other than cement on concrete strength is very true, although not particularly related to the tests under discussion, where there was no opportunity for such variations to occur. Progress in the control of concrete will not be made if we condone lack of uniformity in one factor because other factors also cause variations. Differences in concrete due to proportioning which can be "detected visually" may very well be of much less importance than those which reveal themselves only after considerable elapsed time.

Mr. Wagner's contribution is particularly valuable in that it demonstrates the practical importance of cement variability. It is also of interest that he found low strength levels associated with periods of peak cement production and warm weather. To the extent that peak production was the significant factor, strength variations should be controllable within the manufacturing process. To the extent that the detriment of high temperature to concrete strength was the significant factor, the seasonal variations might well be outside the control of the cement manufacturer. In this connection, however, it should be kept in mind that our tests were conducted under standard temperature conditions and, hence, the variations reflect actual differences in the characteristics of the cements rather than seasonal effects.

AERATION CAUSE OF FALSE SET IN PORTLAND CEMENT*

By W. C. Hansen[1]

Synopsis

Evidence, based on extractions with water and the rates at which cements combine with calcium chloride, is presented to show that cements which develop false set upon aeration do so because the reactivity of the $3CaO \cdot Al_2O_3$ is decreased sufficiently to permit dehydrated gypsum in the cement to precipitate as gypsum in the cement-water paste.

The user of portland cement, except for some special uses, wants a product which, when mixed with aggregates and water, yields a concrete that does not stiffen very markedly during about the first 30 min after mixing and then hardens fast enough to give a fairly high strength in 24 hr. Such a cement is said to have normal setting properties, although normal in this case is not very clearly defined. However, if the concrete stiffens markedly during the first few minutes after mixing, the cement is classed as either flash or false setting.

Calcium sulfate was adopted as a material to grind with portland-cement clinker to prevent flash set. After Rankin and Wright **(1)**[2] reported that the principal compounds in portland cement were $3CaO \cdot SiO_2$, $2CaO \cdot SiO_2$ and $3CaO \cdot Al_2O_3$, technologists **(2, 3)** studied the individual compounds for setting characteristics. These studies showed that a paste of $3CaO \cdot Al_2O_3$ and water released heat at a very rapid rate and stiffened to an unworkable mass within a few minutes. Pastes of the silicates with water released heat and hardened at slow rates. These studies also showed that pastes of $3CaO \cdot Al_2O_3$, gypsum, and water hardened rapidly but that pastes of $3CaO \cdot Al_2O_3$, gypsum, water, and either $3CaO \cdot SiO_2$ or $Ca(OH)_2$ hardened slowly. The results of these studies demonstrated conclusively that $3CaO \cdot Al_2O_3$ was responsible for flash set which was manifested by a rapid rise in the temperature of the cement-water paste and by a pronounced reduction in the workability of this paste within a few minutes after mixing. This workability may be partially restored by remixing the paste of a flash setting cement.

The use of calcium sulfate to prevent flash set led to the development of what has become known as false set. In this type of set, there is no rapid rise in the temperature of the cement-water paste, but there is a pronounced reduction in the workability of the paste within a few minutes. This workability usually can be fully restored by a little remixing of the paste.

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* Presented at the Sixty-first Annual Meeting of the Society, June 22–27, 1958.

[1] Director of Research, Research Laboratories, Universal Atlas Cement Co., Buffington, Ind.

[2] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 1049.

This type of false set was found by various investigators to be caused by crystallization of gypsum from supersaturated solutions. It is possible, under certain conditions of grinding and storing cements, for the gypsum to be either partially or completely dehydrated to either hemihydrate or soluble anhydrite. These compounds readily produce solutions that are supersaturated with respect to gypsum (4).

It seemed that the causes of flash and false set were well known and that it should be possible to explain all cases of false set on the basis of one or both of these causes. However, there is at least one type of false set that has remained unexplained. This is the type in which a cement, as drawn from a silo, will not manifest false setting tendencies, either in the laboratory or in the field, but develops these tendencies when exposed to air for some period of time. In some cases, the bags of cement from a shipment will give satisfactory concrete on a job for several days and then produce false-setting concrete. This paper describes some experiments which appear to provide the explanation for this type of false set.

EXPERIMENTS WITH TYPE IS CEMENT

A portland blast-furnace slag cement was ground commercially with a blend of approximately 60 per cent natural anhydrite and 40 per cent gypsum, which provided an SO_3 content of approximately 2 per cent. The mill operated at about 250 F with a high circulating load which was transported through an air separator with a large volume of air at a relatively low humidity. These are conditions that tend to dehydrate the gypsum. A sample of the cement (identified as sample A) was collected during the grinding and stored in tightly sealed cans until tested. A sample of the cement (identified as sample B) was taken from the silo after a storage period of three weeks. These were subjected to the tests described below.

Three samples of each of the lots of cement were tested. These were as follows:

1. Sample as received,
2. Sample aerated in a thin layer in laboratory air for 24 hr, and
3. Sample aerated in a thin layer in laboratory air for 48 hr.

Extraction Tests:

200 g of cement were shaken with 150 ml of deionized water at room temperature for periods of 1 and 3 min. These pastes were filtered on a suction filter and the extracts were analyzed for CaO, SO_3, and alkalinity. The alkali content of the filtrate was calculated on the assumption that the only materials in the filtrates were alkali and calcium sulfates and hydroxides. Hence,

$$\text{Moles SO}_3 + \text{Moles (OH)}_2 - \text{Moles CaO} = \text{Moles Alkalies.}^3$$

It has been found in many tests in our laboratories that the values for alkalies calculated in this way for such filtrates agree very closely with determined values.

False Set Tests:

Each sample was tested for false set by the ASTM Method for False Set of Portland Cement (C359).[4]

The results of the extraction and false set tests are given in Table I, from which it may be seen that concentrations of alkalies in the filtrates varied between 0.0135 and 0.0311 moles per liter.

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[3] For simplicity, this is expressed as Na_2O.

[4] 1955 Book of ASTM Standards, Part 3, p. 139.

According to data by Hansen and Pressler (5), the concentration of SO_3 in such solutions should be between 0.019 and 0.025 moles per liter when the solid phases are $Ca(OH)_2$ and $CaSO_4 \cdot 2H_2O$. On this basis, the extracts of the "as-received" samples were slightly supersaturated with respect to gypsum at 1 min but were slightly unsaturated at 3 min. On the other hand, the extracts of the samples aerated for either 24 or 48 hr were highly supersaturated at both 1 and 3 min.

TABLE I.—RESULTS OF EXTRACTION AND FALSE SET TESTS ON TYPE IS CEMENT.

Treatment	Results of Extraction Test					Results of False Set Test with 32 per cent Water				
	Time of Shaking, min	Composition of Extract, moles per liter				Penetration, mm				
		SO_3	$(OH)_2$	CaO	Na_2O[a]	Initial	5 min	8 min	11 min	Remix
CEMENT AS-GROUND										
As-Received	1	0.0245	0.0279	0.0286	0.0238	50+	47	46	46	50+
	3	0.0157	0.0433	0.0279	0.0311					
Aerated 24 hr	1	0.0523	0.0238	0.0589	0.0172	47	44	30	16	50+
	3	0.0401	0.0239	0.0458	0.0182					
Aerated 48 hr	1	0.0533	0.0232	0.0621	0.0144	17	14	5	2	48
	3	0.0431	0.0234	0.0512	0.0153					
CEMENT FROM SILO										
As-Received	1	0.0233	0.0285	0.0279	0.0239	50+	45	43	37	45
	3	0.0162	0.0338	0.0276	0.0224					
Aerated 24 hr	1	0.0521	0.0190	0.0560	0.0151	50+	47	46	46	50+
	3	0.0433	0.0242	0.0496	0.0179					
Aerated 48 hr	1	0.0538	0.0225	0.0628	0.0135	46	27	12	10	50+
	3	0.0432	0.0229	0.0515	0.0146					

[a] SO_3 at equilibrium for Na_2O of 0.0135 to 0.0311 varies from about 0.019 to 0.025 moles per liter.

Neither gypsum nor anhydrite will produce solutions that are more than slightly supersaturated with respect to gypsum. Hence, the high degree of supersaturation shown by the aerated samples is evidence that some portion of the gypsum was dehydrated to either hemihydrate or soluble anhydrite during the grinding operation.

Supersaturated solutions of gypsum will not precipitate sufficient gypsum to yield an unsaturated solution. Hence, the unsaturated conditions of the extracts from the "as-received" samples show that calcium sulfate is combining with some phase of the cement at a greater rate than it is going into solution. The only known reaction of this type in a cement paste is the formation of calcium sulfoaluminate (6):

$$3CaO \cdot Al_2O_3 + 3CaSO_4 + xH_2O \rightarrow 3CaO \cdot Al_2O_3 \cdot 3CaSO_4 \cdot xH_2O.$$

It may be seen from Table I that the mortars of the "as-received" samples tended to stiffen slightly during the 11 min of the penetration test. This stiffening, together with the unsaturated condition of the extract at 3 min, indicates that the mixture of gypsum and anhydrite does not dissolve at a sufficiently rapid rate to prevent some tendency toward flash set on the part of the $3CaO \cdot Al_2O_3$. This could be expected, since it is known that natural anhydrite dissolves at a relatively slow rate.

Aeration for 24 hr produced some tendency toward false set, and aeration for 48 hr produced a very strong tendency toward false set. It is well known that the various cement minerals, such as $3CaO \cdot Al_2O_3$ and $3CaO \cdot SiO_2$, are very reactive toward water and carbon dioxide. Hence, it may be expected that the surfaces of these minerals would tend to become coated with reaction products and that these products would retard the rate at which these minerals react with water and calcium sulfate when mixed with water.

It seems impossible for aeration to increase the rate at which the calcium sulfate minerals dissolve in water. Hence, since the aerated samples produced extracts which were highly supersaturated at 3 min with respect to gypsum and the "as-received" samples produced extracts that were unsaturated, it seems that the only conclusion possible is that the reactivity of $3CaO \cdot Al_2O_3$ towards $CaSO_4$ is much greater in the case of the "as-received" samples than in the aerated samples. It appears, therefore, that aeration of the samples, by virtue of inactivation of the $3CaO \cdot Al_2O_3$, produced a condition whereby the dehydrated gypsum formed during grinding could supersaturate the liquid phase of the cement paste and cause false set through the crystallization of gypsum. Accordingly, the conclusion is that the false set shown by the aerated samples is true false set caused by dehydrated gypsum.

These results for false set clearly indicate a reduced reactivity for $3CaO \cdot Al_2O_3$ as the result of aeration. The data of Table I show an Na_2O content of 0.0311 moles per liter for the 3-min extract of the "as-received" sample and only 0.0153 moles per liter for the 3-min extract of the sample aerated for 48 hr. These results indicate a marked reduction in the reactivity of the alkali-bearing phases.

EXPERIMENTS WITH CALCIUM CHLORIDE

Calcium chloride combines with $3CaO \cdot Al_2O_3$ as shown in the following equation (7):

$$3CaO \cdot Al_2O_3 + CaCl_2 + 10H_2O \rightarrow 3CaO \cdot Al_2O_3 \cdot CaCl_2 \cdot 10H_2O.$$

Since calcium chloride is soluble, it is possible to study slurries of cement in calcium chloride solutions (8) and determine the rate at which the chloraluminate is formed. Such a study was made in which two samples of type I cement clinker from the same plant were used. These were ground in a laboratory mill without gypsum to specific surfaces of 1745 and 1785 sq cm per g, respectively, for samples A and B. Each sample was tested as ground, after aeration for 24 hr and after aeration for 48 hr.

The samples were tested as slurries of 200 g of ground clinker with 150 ml of a solution of calcium chloride made up to contain 3.0 per cent Cl_2 by weight of solution, which would be equivalent to 2.25 per cent Cl_2 by weight of cement. These slurries were shaken at room temperature and were filtered on a Buchner funnel after prescribed shaking periods of 1, 5, 15, and 30 min, and the filtrates were analyzed for chloride. It required a filtering time of less than 1 min to obtain sufficient filtrate for the analysis. The results of these analyses are given in Table II.

The data of Table II show that aeration markedly reduces the rate at which calcium chloride reacts with cement and substantiate the conclusion stated above, on the basis of data of Table I, for SO_3 and alkalies, to the effect that aeration tends to decrease the reactivity of the $3CaO \cdot Al_2O_3$.

TABLE II.—EXTRACTION DATA FOR SAMPLES OF CLINKER IN SOLUTIONS OF CALCIUM CHLORIDE.

Sample Type and Treatment	Grams of Cl_2 Reacted per 100 g of Clinker for Time Indicated			
	1 min	5 min	15 min	30 min
Sample A:				
As-Ground	0.22	0.28	0.34	0.36
Aerated 24 hr	0.08	0.14	0.22	0.24
Aerated 48 hr	0.03	0.11	0.22	0.26
Sample B:				
As-Ground	0.20	0.27	0.31	Set
Aerated 24 hr	0.05	0.11	0.20	0.25
Aerated 48 hr	0.02	0.14	0.18	0.22

TABLE III.—FALSE SET IN QUARTZ PASTES AS DETERMINED BY ASTM METHOD C 359.[a]

SO_3, per cent	Paste Containing Gypsum, Penetration, mm					Paste Containing Molding Plaster, Penetration, mm				
	Initial	5 min	8 min	11 min	Remix	Initial	5 min	8 min	11 min	Remix
0	50+	45	46	44	50+	50+	45	46	44	50+
0.25	47	43	42	42	47	49	40	38	30	50+
0.50	46	44	40	38	46	46	38	27	20	50+
1.00	46	45	41	43	47	48	10	8	7	50+
1.50	47	47	42	40	46	47	5	0	0	50+
2.00	48	44	41	41	48	41	0	0	0	50+

[a] Tentative Method of Test for False Set of Portland Cement (C 359 – 56 T), 1955 Book of ASTM Standards, Part 3, p. 139.

So far as is now known, the reactions of calcium sulfate and calcium chloride in a fresh cement paste are as follows:

$$3CaO \cdot Al_2O_3 + 3CaO \cdot SO_3 + xH_2O \rightarrow 3CaO \cdot Al_2O_3 \cdot 3CaO \cdot SO_3 \cdot xH_2O$$

$$3CaO \cdot Al_2O_3 + CaCl_2 + xH_2O \rightarrow 3CaO \cdot Al_2O_3 \cdot CaCl_2 \cdot xH_2O$$

These equations show that 3 moles of SO_3 combine with $3CaO \cdot Al_2O_3$ whereas only one mole of Cl_2 does. The molecular weights are 80.07 and 70.91 for SO_3 and Cl_2, respectively. Hence, 70.91 g Cl_2 is equivalent to $3(80.07)SO_3$ or 1 g is equivalent to 3.38 in combining with $3CaO \cdot Al_2O_3$. The 0.22 g of Cl_2 that combined in 1 min (Table II) with 100 g of clinker is therefore equivalent to 0.74 g of SO_3. Normally, cements contain from about 1.5 to 2.5 g SO_3 per 100 g. Hence, it appears that, under certain conditions, approximately 30 to 50 per cent of the SO_3 in a type I cement paste may be combined as calcium sulfoaluminate within the first one or two minutes. Hansen and Hunt (9) give data that show most of the SO_3 in cements is combined within 12 to 18 hr.

The molecular weight of $3CaO \cdot Al_2O_3$ is 270 and that of $3SO_3$ is 240. Hence, 1 g of SO_3 will combine with 1.1 g of $3CaO \cdot Al_2O_3$. The type I clinker used in this study had a calculated $3CaO \cdot Al_2O_3$ content of approximately 13 per cent. The 0.74 g SO_3 calculated above is equivalent to about 0.8 g $3CaO \cdot Al_2O_3$ or approximately 7 per cent of the $3CaO \cdot Al_2O_3$ in the clinker.

EXPERIMENTS WITH QUARTZ POWDERS

Three samples of quartz sand were ground in a cold laboratory mill to finenesses comparable to those of portland cements. The first sample contained no calcium sulfate; the second contained gypsum equivalent to 2.0 per cent SO_3;

and the third contained molding plaster, $CaSO_4 \cdot O \cdot 5H_2O$, equivalent to 2.0 per cent SO_3. The first and second were blended to powders having SO_3 contents as gypsum from 0 to 2.0 per cent in 0.25 per cent increments and the first and third were blended to give SO_3 contents as plaster of Paris from 0 to 2.0 per cent in 0.25 per cent increments. The results for false set by ASTM Method C359 are given in Table III.

The data of this table show that the 30 per cent water required for this test was only sufficient to yield penetrations between 38 and 50 in the pastes made with the gypsum-bearing quartz powder. They also show that the test indicated a little false set for the sample containing 0.25 per cent SO_3 as molding plaster and that, even with 0.50 per cent SO_3 as molding plaster, the false-setting tendencies were not marked. These results indicate that a cement could contain up to about 0.50 per cent SO_3 as dehydrated gypsum without showing marked false-setting tendencies.

DISCUSSION OF RESULTS

Blanks and Gilliland (10) state: "Frequently, however, concrete afflicted by false set in the cement will escape detection on the job. This may result from extended periods of mixing or agitation that break through the stiffening."

The ease with which false set can be eliminated by mixing makes it difficult to correlate results of laboratory tests with performance in the field. Hence, the results of laboratory tests cannot be used for very definite conclusions on field performance. However, the results of the studies reported in this paper indicate that cements can contain a relatively large proportion of their calcium sulfate in the form of either hemihydrate or soluble anhydrite and not show false set if they are not exposed to moisture and carbon dioxide to any appreciable extent prior to use. The amount of dehydrated gypsum that will not cause false set should vary with the $3CaO \cdot Al_2O_3$ content of the cement. It appears from the calculations made in this study that about 0.5 per cent of SO_3 would be combined in most type I and type III cements within the first one or two minutes after mixing with water. Also, it appears that it requires about 0.5 per cent to produce false set. Hence, it appears that such cements might contain at least 1.0 per cent SO_3 as dehydrated gypsum without showing false set, provided they are used with practically no exposure to moist air.

Acknowledgment:

The author takes pleasure in acknowledging the assistance of his colleagues who carried out the experimental work of this project.

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DISCUSSION

Mr. Myron A. Swayze[1] (*presented in written form*).—The development of false set due to aeration of cements which show perfectly normal sets when freshly ground is a matter which concerns every cement manufacturer. It is highly disconcerting to any producer to make and ship cements which are free from this annoying property, only to find it developing in sacked cement in dealers' warehouses or to have it induced by contact with moisture-laden compressed air in the transfer through air pumps from bulk cars to the users' bins.

To arrive at a better understanding of causes for this development of false set by aeration, the Lone Star Cement Research Laboratory has conducted a series of research tests on the subject. In order to avoid a quick setting action with no retarder addition, a sulfate-resistant type III plant clinker with the following composition was selected for these tests:

C_3S	73.9	Free CaO	0.6
C_2S	9.9	MgO	0.6
C_3A	None	SO_3	0.01
C_4AF	11.2	Alkalies	0.04
C_2F	3.7	Loss	0.08

Grinding was done in a laboratory mill at room temperature of this clinker by itself, and with additions of gypsum, calcined gypsum, or anhydrite. These grinds were made dry. Duplicate grinds were then made of clinker alone and with gypsum and anhydrite in which 0.2 to 0.5 per cent water was used as a grinding aid. The dry grind with calcined gypsum coated the mill so badly that frequent dumping to clean the grinding media was necessary. In consequence, the final product was already in an aerated condition when grinding was finally completed. The bad false set tests on this sample are therefore not reported here.

All grinds with retarder contained approximately 2.5 per cent SO_3. The fineness of the completed grinds as determined by Wagner turbidimeter was approximately 2700 sq cm per g.

Additional grinds were made with Ottawa silica sand instead of clinker. Enough gypsum, calcined gypsum, or anhydrite was added to provide 2.5 per cent SO_3 in each mix.

All of these samples were subjected to the ASTM False Set Test (Method C 359)[2] after the following types of exposure:

1. Fresh sample as discharged from mill,
2. Fresh sample aerated 16 hr in air at 60 per cent relative humidity,

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[1] Director of Research, Lone Star Cement Corp., New York, N. Y.

[2] 1955 Book of ASTM Standards, Part 3.

TABLE IV.—FALSE SET TESTS ON COLD GROUND LABORATORY CEMENTS.
ASTM Method C 359, 32 per cent water in mortar

Sample Condition	Penetration, mm					Penetration, mm				
	Initial	5 min	8 min	11 min	Remix	Initial	5 min	8 min	11 min	Remix
CLINKER ONLY										
	Dry Grind					0.5 Per Cent Water				
Fresh sample	50	50	50	47	50	50	50	50	50	50
Aerated 16 hr	50	1	0	0	50	50	50	49	12	50
Heated 260 F, 7 days	50	1	0	0	50	50	50	42	5	50
Aerated 16 hr	50	11	0	0	50	50	50	33	2	50
CLINKER PLUS GYPSUM										
	Dry Grind					0.5 Per Cent Water				
Fresh sample	50	50	50	50	50	50	50	50	50	50
Aerated 16 hr	50	3	0	0	50	50	2	1	1	50
Heated 260 F, 7 days	50	0	0	0	50	50	0	0	0	50
Aerated 16 hr	50	0	0	0	50	50	0	0	0	50
CLINKER PLUS ANHYDRITE										
	Dry Grind					0.2 Per Cent Water				
Fresh sample	[a]50	50	5	2	50	50	50	50	50	50
Aerated 16 hr	50	50	2	0	50	50	50	2	0	50
Heated 260 F, 7 days	50	39	3	0	50	50	50	38	3	50
Aerated 16 hr	50	50	2	0	50	50	50	50	35	50

[a] Partial aeration during grinding.

TABLE V.—FALSE SET TESTS ON COLD GROUND SAND-RETARDER GRINDS.
ASTM Method C 359, 32 per cent water in mortar

Sample Condition	Penetration, mm					Penetration, mm				
	Initial	5 min	8 min	11 min	Remix	Initial	5 min	8 min	11 min	Remix
	Sand-Gypsum: 2.54 SO_3					Sand-Anhydrite: 2.44 SO_3				
Fresh sample	50	50	50	50	50	50	50	50	50	50
Aerated 16 hr	50	50	50	50	50	50	50	50	50	50
Heated 260 F, 7 days	2	0	0	0	18	50	50	50	50	50
Aerated	35	0	0	0	18	50	50	50	50	50
	SAND-PLASTER: 2.44 SO_3									
Fresh sample	48	48	48	46	50					
Aerated 16 hr	4	20	26	20	50					
Heated 260 F, 7 days	48	48	47	48	50					
Aerated	17	10	9	7	50					

3. Fresh sample heated 7 days at 260 F, cooled in can, and

4. Heated sample aerated 16 hr in air at 60 per cent relative humidity.

Considering first the results on false set of the cements in Table IV, it will be noted that for the grinds, with or without water, of straight clinker, the freshly ground samples had no false set tendency. Aeration of the sample ground dry caused a strong false set to develop, as did the heating treatment and aeration of the heated sample. For the sample in which water was used as a grinding aid, aeration of the fresh sample, heating, and aeration of the heated sample induced only a moderate amount of false set. Since no calcium sulfate in any form was present in either of these grinds, the false set cannot be attributed to dehydrated gypsum but must be ascribed to some component in the cement clinker which was affected by aeration.

Both grinds containing gypsum were completely free from false set when fresh and both developed an exaggerated false set after aeration at room temperature. Again, since there had been no dehydration of gypsum during grinding, the cause of false set on aeration must be looked for in the clinker component. Heating of the cement from both these grinds must have converted the gypsum largely to hemihydrate, and the strong false set caused by heating and subsequent aeration of the heated sample can unquestionably be attributed to dehydrated gypsum.

The dry clinker-anhydrite grind caused a bad coating condition in the mill which required the charge to be dumped several times to clean the grinding media. This induced a moderate amount of false set by preaeration of the fresh sample. Further aeration, heating, and reaeration of the heated sample did not materially change this false set condition.

The clinker-anhydrite mix ground with 0.2 per cent water had no sign of false set when freshly ground. Aeration of this cold ground sample induced false set in 8 min. Heating of the fresh sample induced a moderate false set which may have come from the 17 per cent gypsum content in the anhydrite. Aeration of the heated sample restored it to a practically normal set.

To understand the possible contribution of various forms of calcium sulfate to false set in portland cement requires some knowledge of the different forms of artificially dehydrated gypsum. I understand that to obtain stable sets in molding plaster the calcination temperature must be carried up to 300 F. At lower temperatures, set is not only much faster, but unstable. In consequence, to pass the ASTM set requirements of 20 to 40 min as given in ASTM Specification C 59 – 50,[2] final calcination temperatures are quite generally held to 300 to 305 F. Such plaster is entirely too slow setting for manufacture of wall board, in which the plaster must set within the 5 to 6 min allowed by the continuous process. This is accomplished by intermixing small quantities of finely ground gypsum and potassium sulfate with the plaster, both of which act as accelerators.

Another hair-trigger form of calcined gypsum is artificial anhydrite formed by calcining gypsum at 380 F. This form of anhydrite is so avid for water that it is a good desiccating agent. Its set is very fast. However, its temperature of formation is above the range of normal cement grinding.

Table V shows false set tests on grinds of sand and the three forms of calcium sulfate tested. The molding plaster used was calcined in a gypsum plant kettle at 305 F and contained no added retarder or accelerator.

The behavior of gypsum, cold ground with sand, is as one might expect.

Neither the fresh or aerated fresh sample had any sign of false set. The sample heated to 260 F was so fast setting that practically full stiffening of the paste occurred in the 60 sec between the end of mixing and the time for the initial penetration test. Aeration of this heated sample slowed down the rate of set slightly, but the final result was the same.

The anhydrite ground with sand caused no false set due to heating or to aeration of either fresh or heated samples.

The molding plaster-sand grind showed no false set in the fresh or heated grinds. This is not surprising in view of its original higher calcination temperature and its Vicat set of 35 min, which should not be disturbed by reheating at a lower temperature. The acceleration of this slow set by aeration, however, is somewhat surprising. Conversion of some of the hemihydrate to gypsum by moisture absorption could explain the change, but this was apparently denied by the fact that aeration did not increase loss on ignition. We have no explanation of this behavior. Aeration of dry grinds containing clinker usually increased losses by 0.2 to 0.3 per cent.

With all the evidence here given pointing away from deactivation of C_3S and C_3A grains by aeration which would allow development of false set through uncontrolled recrystallization of dehydrated gypsum, the question then arises as to what compound in cement clinker is responsible for the change in setting characteristics following aeration.

The clinker used for our tests contained no C_3A, with the total of C_3S, C_2S, and a particularly inert form of the ferrite phase constituting 98.7 per cent of the clinker. SO_3 and alkali contents were little more than traces. C_2S and the iron phase are known to react very slowly with water, leaving only the C_3S as the compound to be suspected.

Nearly all portland cements can be changed from normal set to a false setting condition by a few hours of aeration at moderately high humidities. From observation over many years we believe this change is due to a temporary activation of C_3S particles by absorption or partial combination with very thin films of water vapor on the grain surfaces. These freshly activated surfaces are ready for very rapid combination with more water when mixed with it and a gelling action develops quickly. Our belief is strengthened by the following observations:

1. Cements with high C_3S contents are more susceptible to false set development on aeration than those with lower C_3S contents. The amounts of C_3A present have little or no influence on the intensity of the false set action, and

2. False set induced by exposure to moist air is a transient phenomenon. Bad cases of false set arising from this exposure can be "cured out" either by prolonged exposure in air of normal humidity or by storage in sealed containers. The deactivation theory cannot explain this change of action. The idea of temporary activation of C_3S grains by moisture can explain it, since the active surfaces become inactive as soon as they combine with the moisture present to form a thin shell of stable hydration products.

MR. W. C. HANSEN (*author*).—I would like to make a few comments regarding Mr. Swayze's discussion. I do not believe that there is any argument between Mr. Swayze and me. He is describing an entirely different type of false set caused by aeration than I am. I believe that he agrees that the type of false set that I described is caused by dehydrated gypsum. We get one type of false set when a cement containing dehydrated gypsum is aerated.

There is another kind which Mr. Swayze has described in his paper. It appears that the tricalcium silicate can be activated by aeration so as to produce false set. We are talking about two entirely different types of false set which are both caused by aeration and Mr. Swayze's paper might have been a paper in its own right instead of a discussion of mine.

MR. SWAYZE.—I agree thoroughly with the author. I did not mean that his theories on dehydration are wrong. I think it is the principal source of false set, but we do have this other effect of aeration on cements with moderate or high C_3S contents. It is another sort of a false set caused by adsorption of moisture on the surfaces of C_3S.

MR. STANTON WALKER.[3]—I wish to ask a question concerning false set. In the ready-mixed concrete industry we hear a lot about false set, flash set, and quick stiffening in general. In our industry the mixing time is fairly long, almost always more than 15 or 20 min and frequently longer than that. I have been told by many of my friends in the cement industry that we should not worry about false set since any manifestation of it will disappear in a period of time less than our normal mixing time of a minimum of, say, 15 min. I would like to know if there is agreement with that advice.

MR. HANSEN.—I think that Mr. Walker is right. That is, if concrete is mixed for several minutes, there will be no manifestation of false set. The troubles with false set occur on the jobs in which the concrete is mixed for a short period of time and is allowed to stand for some period of time in the mixer, in forms, or on the subgrade before the final placing. Under these conditions, concrete made with a false-setting cement will stiffen and require additional working for proper placing. ASTM Method C 359 is based on the experience that false set will not develop unless the mixing periods are relatively short and will be destroyed by remixing.

I know that there are men in the concrete industry who believe that false set causes the use of greater amounts of water and, accordingly, decreases strength and durability. I am not certain that this is true if the concrete is placed and worked into the final form immediately after mixing is stopped or, if it is remixed within a few minutes after the first mixing period.

MR. WALKER.—I would like to ask the author this question. Of what are we getting a manifestation when this quick stiffening occurs after a mixing time of more than 15 or 20 min—with this not restricted to hot weather alone?

MR. HANSEN.—I do not think that it is due to the false set that Mr. Swayze and I have been talking about. It is the natural stiffening of the portland cement caused by the normal reactions of the cement with water. Mr. Swayze has shown in his paper that portland cement is a very reactive chemical and that a little aeration can affect the tricalcium silicate. The cement minerals can react with water immediately upon mixing with water and, in a period of 15 min there should be an appreciable amount of reaction. The stiffening caused by this reaction is normal and should not be classed as false set.

MR. SWAYZE.—I might comment further on Mr. Walker's question. Suppose cement has been ground at a very high temperature so that the gypsum is completely dehydrated. Then you should have a stable slow set plaster in the cement which will not begin to set until about the time you get ready to dump the concrete out of your truck. You might get a false set from that case, but that will be exceedingly rare.

In the manufacture of wallboard,

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[3] Director of Engineering, National Sand and Gravel Assn., Washington, D. C.

gypsum producers use seed crystals of very finely ground gypsum, and alkali sulfates as accelerators, and then the set, instead of taking place in 30 or 35 min, will start within a minute or two. By the time the wallboard reaches the end of the line, the set is completed. However, in cement if you have a completely dehydrated gypsum due to very high grinding temperatures, you still have the clinker portion to consider. The alkali sulfates there are quickly soluble and will produce both gypsum and alkali by reaction with hydrated lime which will act as accelerators of this long-retarded gypsum or hemihydrate. The author has answered the other part of your question: the finely ground cements are more active than the other ones. Ready-mix contractors always give the cement producers some help in the way of cement fineness. After rotating the cement and aggregate for a half or three-quarters of an hour in a truck mixer, a considerable amount of grinding takes place, so that you have a finer cement than you started out with.

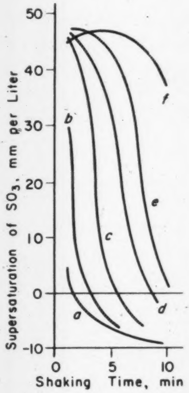

FIG. 1.—Typical Curves of Degree of Supersaturation of SO_3 in Extraction Test.

a, b, and *c* are shown by normal settings cement; *d, e,* and *f* are by false setting cement.

MR. TOSHIO MANABE[4] (*by letter*).—I have studied in recent years about the mechanism of false set as it appears in aerated portland cements. I would like to discuss the probable mechanism of false set.

Extraction Tests (Degrees of supersaturation with respect to gypsum):

The procedures were essentially the same as those described by the author but differed from them in water cement ratio and shaking periods. (100 g of cement were shaken with 50 ml of distilled water at 20 C for periods of 1, 3, 5, 7, and 10 min).

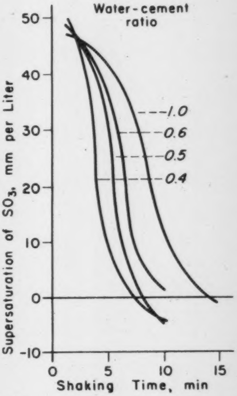

FIG. 2.—Effect of Water Content upon the Degree of Supersaturation of SO_3.

The degrees of supersaturation of SO_3 were calculated from the results obtained and the solubility curve of $CaSO_4 \cdot 2H_2O$ and $Ca(OH)_2$ in dilute alkali solution determined by Hansen and Pressler.[5] A few typical results of the degrees of supersaturation of SO_3 are given in the accompanying Fig. 1. In a fresh cement, which does not manifest false setting tendencies, the degrees of supersaturation are generally represented by curve *a* or *b*. When the cement is aerated, the supersaturation curve shifts from *a* to *b*, and then to curves *c*, *d*, *e*, and to *f*, successively. If the cement is further aerated at the high humidity, the supersaturation curve turns back to *d* or *c*.

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[4] Research Member, Chemical Section, Research Laboratory, Nihon Cement Co., Ltd., Tokyo, Japan.

[5] See reference (**5**) of author's paper.

In relation to the results of penetration tests with neat cement paste (by Federal Specification), it is found that the cement, which shows the supersaturation curve *a*, *b*, or *c*, has normal setting properties, but the cement represented by the curve *d*, *e*, or *f* manifests false setting tendencies. In other words, when the extracts at 5 min are still highly supersaturated, the cement shows the false setting tendencies. If the mixing period is 2 min instead of 3 min, the cement in curve *c* manifests also false set, and on the other hand the cement in curve *d* does not show false setting tendencies if it is mixed for 4 min.

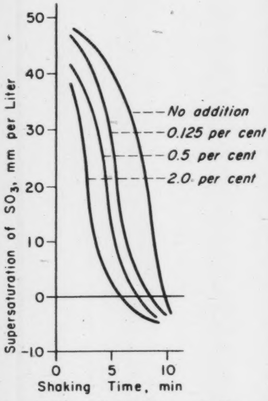

FIG. 3.—Effect of Addition of Gypsum upon the Degree of Supersaturation of SO_3.

Since the water content in penetration tests is about 25 to 30 per cent and different from that in extraction tests, the effect of water content on the results of extraction tests must be considered. From Fig. 2, which illustrates the effect of the water-cement ratio, it is clear that as the water content decreases, the time at which the degrees of supersaturation of SO_3 begin to decrease rapidly comes quickly. From the above tendencies, it may be seen that the time at which the degrees of supersaturation of SO_3 begin to decrease rapidly, corresponds to the stiffening time. From the results reported by Birss and Thorvaldson,[6] it is seen that the rapid decreasing period of high supersaturation of SO_3 corresponds to the precipitating period of gypsum. Accordingly, the stiffening phenomenon of false setting cement paste may be caused by the precipitation of gypsum which takes place after mixing.

By the addition of a small amount of finely pulverized gypsum to the false setting cement, the false set is sometimes relieved. Figure 3 gives the supersaturation curves in such a case. By the addition of 0.125 per cent gypsum, the cement still manifested the false setting tendencies in penetration test. With 0.5 per cent addition, it became the normal setting cement, but if the paste was mixed for 2 min, it showed the false setting tendencies.

Rate of Combination of SO_3 *as Calcium Sulfoaluminate:*

Gypsum is soluble, whereas the calcium sulfoaluminate is almost insoluble in lime solution, Forsen,[7] using this difference in solubility, investigated the amount of combined gypsum in cement paste by extraction with lime solution. However, this method is not utilized in determining the amount of combined gypsum during the first few minutes of initial hydration period because a further combination takes place during the process of the extraction.

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[6] F. W. Birss and T. Thorvaldson, *Canadian Journal of Chemistry*, Vol. 33, p. 870 (1955).

[7] Forsen, "The Chemistry of Retarders," and Accelerators," *Proceedings*, Symposium on Chemistry of Cement, Stockholm, p. 298 (1938).

I have developed a new method using radioisotope sulfur-35. The procedures are briefly shown as follows: Paste, made by mixing 1 g of cement and 0.5 ml of distilled water, is extracted with 100 ml of lime solution containing SO_3 labelled with sulfur-35 after each time-period. The amounts of SO_3 in the lime solution before and after extraction are analyzed, and the specific radio activities of SO_3 in both solutions are also counted.

Now, let W_c represent the amount of SO_3 in the cement, W_b the amount of SO_3 combined with aluminate (W_b is the unknown), and W_e, the amount of SO_3 in the lime solution before extraction. When the paste is extracted with such a lime solution, uncombined SO_3 ($W_c - W_b$) is dissolved so that the amount of SO_3 in the extract is ($W_e + W_c - W_b$). Some part of it would combine with aluminate during the extraction, but if it is assumed that uncombined SO_3 ($W_c - W_b$) would be dissolved in a moment, the balance of specific activities may be represented by:

$$Se \cdot We = S(We + Wc - Wb)$$

Hence, combined SO_3 (W_b) is given by:

$$W_b = (W_e + W_c) - W_e \cdot S_e / S$$

where:

S_e and S are specific activities of SO_3 in the lime solutions before and after extraction, respectively.

The results obtained are illustrated in Figs. 4, 5, and 6, and summarized briefly as follows: (*a*) When cement which has normal setting properties is mixed with water, the considerable amounts of gypsum are combined in calcium sulfoaluminate in a few minutes and then the rate of its formation becomes very slow (Fig. 4, curves *A*, *B*, and *C*), (*b*) whereas, in slightly aerated, false setting cement, the combination of gypsum occurs about 3 min later, that is, the combination of gypsum with aluminate is prevented for some reason (Fig. 4, curves *A'*, *D*, and *E*), and (*c*) when cement is aerated at high humidity, combination of gypsum takes place also

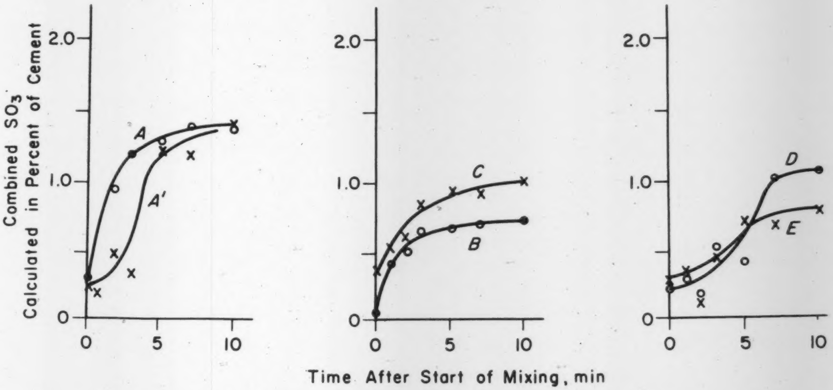

FIG. 4.—Amount of Combined SO_3 During the First Few Minutes of Initial Hydration Period.
A, *B*, and *C* are normal setting cements.
A,' *D*, and *E* are false setting cements.

(Fig. 5). In paste made from such a cement, combined SO_3 decreases at first, and then increases rapidly (Fig. 6).

Probable Mechanism of False Set:

By adding ground clinker to plaster of Paris, the stiffening time of gypsum is accelerated; that is, clinker accelerates the stiffening of gypsum.

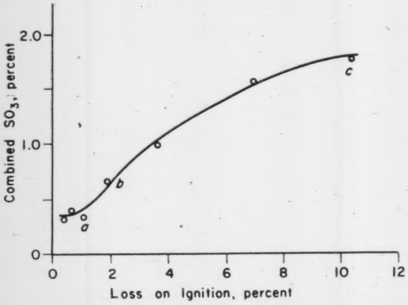

FIG. 5.—Amount of Combined SO_3 in Cement, Aerated Under Atmosphere of High Humidity. (Relative humidity 90 to 100 per cent.)

In the case of cement, the gypsum in cement is dehydrated to hemihydrate or soluble anhydrite during grinding. The stiffening time of such a dehydrated gypsum occurs within 3 min (mixing time) by the accelerating action of clinker, so that false setting tendencies are not observed. But the accelerating action of clinker is weakened by the aeration, that is, the stiffening time occurs after 3 min and the false setting tendencies are manifested.

The manner in which the clinker acts as an accelerator of dehydrated gypsum is not clear, but it is likely that the crystals of calcium sulfoaluminate formed by the combination of aluminate with gypsum behave as crystal nuclei of precipitation of gypsum. Due to the aeration, the surfaces of the clinker may be coated with a thin film of aerated products, and alkali carbonate may be also produced. These products would prevent the formation of calcium sulfoaluminate, then the precipitation of gypsum is retarded and the false setting tendencies can be observed.

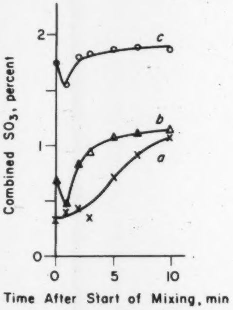

FIG. 6.—Amount of Combined SO_3 During the First Few Minutes of Initial Hydrate Period, in the Case of Cements Aerated at High Humidity. (Shown *a*, *b*, and *c* in Fig. 5.)

It is also attributed to their behaviors as crystals nuclei that false set is relieved by the addition of small amounts of finely crystalline gypsum to false setting cement.

MR. HANSEN.—Mr. Manabe is to be congratulated on the development of a new method of studying the rate of combination of calcium sulfate in cement pastes.

REFLECTANCE TESTS FOR CONCRETE CURING MATERIALS*

BY C. C. RHODES,[1] M. H. JANSON,[1] AND M. G. BROWN[1]

SYNOPSIS

Stress control through the regulation of early temperature changes in concrete is an important function of the curing operation. Since the primary purpose of using white curing materials is to reduce heat pickup from solar radiation, the measurement of reflectance is of considerable significance in procuring such materials. Because existing preference is divided between the luminous reflectance method of ASTM Method E 97[2] and the infrared reflectance method of Federal Specification TT-P-141b, this study was undertaken to provide a sound basis for evaluation of these two methods and development of reflectance tests in general.

Three factors enter into the development of a significant and practical reflectance test for concrete curing materials: (1) spectral distribution of solar radiation received at the earth's surface, (2) suitability of various photometers for measurement of reflectance in the most significant spectral range, and (3) spectral characteristics of the curing materials. An analysis of these three factors indicates that measurements in the visible range are as significant as those in the infrared and that ASTM Method E 97 for routine testing and ASTM Methods D 307 and D 791[3] for reference are the best that can be found at present using commercial photometric equipment currently available at a reasonable cost.

It has long been recognized that stress control through the regulation of early temperature changes in concrete is an important function of the curing operation. Since the principal purpose of using white materials of either the sheet or membrane-forming type is to reduce both the rate and amplitude of temperature change due to absorption of solar radiation, the specification and measurement of reflectance are of considerable significance in procuring such materials.

Probably the method most commonly used in the past for measuring reflectance of white curing materials is ASTM Method E 97, Method of test for 45-deg, 0-deg Directional Reflectance of Opaque Specimens by Filter Photometry.[2] There seems to be a rather prevalent belief, however, that a reflectance test for curing materials should measure infrared rather than luminous reflectance, and it has been proposed that the infrared method be incorporated in ASTM specifications for white curing paper. Thus there is a need for clarification of the principles involved and their application to reflectance specifications and tests for curing materials.

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* Presented at the Sixty-first Annual Meeting of the Society, June 22–27, 1958.

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[2] Method of Test for 45-deg, 0-deg Directional Reflectance of Opaque Specimens by Filter Photometry (E 97 – 55), 1958 Book of ASTM Standards, Parts 4, 6, 8.

[3] Method of Test for Spectral Characteristics and Color of Objects and Materials (D 307 – 44), 1958 Book of ASTM Standards, Part 8.

Method of Test for Luminous Reflectance, Transmittance, and Color of Materials (D 791 – 54), 1958 Book of ASTM Standards, Part 9.

The purpose of this paper, therefore, is twofold: (*a*) to discuss some of the more important facts and principles which should govern the development of reflectance tests for curing materials; and (*b*) to apply these facts and principles to the design of the best test possible within the limitations of currently available instruments and spectral characteristics of the materials themselves. In carrying out this purpose the discussion which follows is divided among four major topics: (1) fundamental concepts and data, (2) reflectometric methods

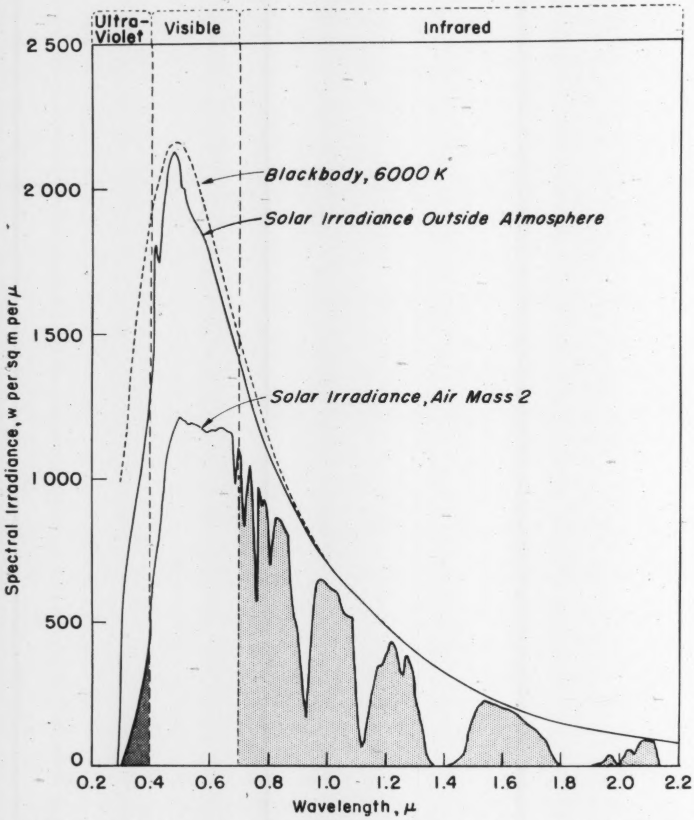

FIG. 1.—Spectral Distribution of Radiant Energy from the Sun and a Blackbody Radiator at 6000 Kelvin.

and instruments, (3) spectral characteristics of curing materials, and (4) recommended test.

FUNDAMENTAL CONCEPTS AND DATA

Throughout the following discussion, the first and most important basic fact to remember is that, in general, all radiation is converted to heat when absorbed by a body on which it falls. Exceptions to this rule, such as fluorescence and the production of photochemical and photoelectric effects, need not be considered here. It follows, therefore, that equal amounts of radiant energy, in whatever portions of the spectrum they may occur, produce equal quantities of heat when totally absorbed. Choosing an interval of the spectrum, then, in which to make reflectance tests for curing materials depends largely on the spectral distribution of solar radiation reaching the earth's surface (insolation). Furthermore, the particular wavelength band selected should include as great a portion of the total insolation as possible. This being the case, it will be profitable to examine the composition of solar radiation rather closely. Since a knowledge of reflectometric terms is also necessary for an understanding of the significance of the measurements to be made, this aspect of the subject will also be presented briefly.

TABLE I.[a]—CALCULATED SOLAR IRRADIATION AT SEA LEVEL.

Wavelength, μ	Energy, w per sq m					
	Air Mass = 0	Air Mass 1	Air Mass 2	Air Mass 3	Air Mass 4	Air Mass 5
0.29 to 0.40	94.6	40.1	19.8	10.0	5.4	2.7
0.40 to 0.70	540.0	419.7	327.8	258.6	205.8	163.7
0.70 to 1.1	365.4	309.2	267.5	233.4	205.1	181.5
1.1 to 1.5	162.5	95.3	70.4	57.0	48.1	40.7
1.5 to 1.9	72.8	50.8	45.1	41.0	38.0	35.2
1.9 to infinity	86.8	12.8	9.2	7.5	6.5	5.8
Total	1 322.1	927.9	739.8	607.5	508.9	429.6

[a] Taken from P. Moon, "Proposed Standard Solar-Radiation Curves for Engineering Use," *Journal of the Franklin Institute*, Vol. 230, July–Dec., 1940, p. 583.

Solar Radiation:

Solar radiation at normal incidence on the earth varies widely because of variations in the sun itself, variations in the distance from sun to earth, differences in atmospheric scattering, and differences in atmospheric absorption. As a convenient reference for comparison with irradiation at the earth's surface, the spectral distribution of solar energy outside the atmosphere plotted from data by Moon (**1**)[4] is shown in Fig. 1. This curve corresponds closely to that of a blackbody radiator at 6000 Kelvin at wavelengths beyond about 1.25 μ, but is depressed at the shorter wavelengths due to selective absorption in the sun itself.

Next, let us see what happens to this radiation by the time it reaches the earth's surface. Direct radiation from the sun is scattered by molecular nitrogen, oxygen, water vapor and other gases, and by dust. The degree of scattering is a function of wavelength and increases exponentially as the ultraviolet end of the spectrum is approached. This phenomenon gives rise to the characteristic blue color of the sky on a clear day. Absorption by the atmosphere modifies the amount and spectral distribution of solar radiation still further. Ozone absorbs selectively in the ultraviolet, and water vapor and carbon dioxide absorb heavily in the infrared. The net result of atmospheric effects is shown in the third curve of Fig. 1, which gives the spectral irradiance for air mass 2, that is, for a path through the atmosphere twice its length at normal incidence.

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[4] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 1071.

Additional data from Moon (1) are given in Table I, wherein are listed results for five values of air mass, thus making possible the calculation of irradiation at any hour of the day except near sunrise or sunset. He suggests that the curve for air mass 2 be used whenever a single, standard solar-radiation curve is needed in engineering calculations for places near sea level. Values for air mass 2 are sufficiently representative for the present purpose and will be assumed in all subsequent discussion unless otherwise noted.

As may be seen from the curves of Fig. 1 and data of Table I, almost 100 per cent of the sun's energy falls within the wavelength range of 0.3 to 2.2 μ. Of this range, the interval of approximately 0.3 to 0.4 μ constitutes the ultraviolet portion, 0.4 to 0.7 μ the visible, and 0.7 to 2.2 μ the infrared. The entire range of wavelengths beyond 2.2 μ contributes less than 2 per cent of the total insolation. However, the energy level is much higher in the visible portion of the spectrum and falls off quite rapidly with increasing wavelength in the infrared. There is also considerable absorption by the atmosphere in the infrared region so that the total energy, exclusive of the ultraviolet, is made up of almost equal portions of visible and infrared radiation. The amount of energy in the ultraviolet band is relatively small, only 4 per cent of the total, and is of little significance in the present instance.

Reflectometric Terms and Definitions:

It is well known that illuminated objects do not appear equally bright from all directions, and certain conditions and terms must be defined in order to understand the significance of reflectance measurements. Three quantities are of primary importance, namely, reflectance, specular reflectance, and directional reflectance.

Reflectance is defined as the ratio of the total radiant energy reflected from an object to the total radiant energy incident on it, and will vary with the angular distribution of the incident energy (2,3). Since no object can reflect more energy than falls on it, the value of reflectance cannot exceed unity.

Specular reflectance is the ratio of reflected to incident energy in the direction of mirror reflection only. Its value will depend on the angle of incidence and, again, cannot exceed unity. Gloss and sheen are examples of the manifestation of luminous specular reflectance, being defined by particular angles of illuminating and viewing.

Directional reflectance is the ratio of the radiance that the surface has under any specified irradiating and receiving conditions to the radiance that a perfectly diffusing, perfectly reflecting surface would have under the same conditions. Because directional reflectance is a ratio of the reflectance of two surfaces under any specified set of conditions, its value may be either greater or less than unity. The primary standard of reflection is freshly prepared magnesium oxide, MgO, which has a (total) luminous reflectance of more than 0.97 and a luminous directional reflectance of 1.00. Its total reflectance falls off somewhat in the infrared, reaching a value of 0.94 at 2.2 μ. Directional reflectance depends on the spectral and angular distribution of the incident energy and the direction of reception. The angles generally specified for irradiation and reception are 45 and 0 deg to the normal, respectively.

The terms defined above are general for radiation of all wavelengths. When the incident energy is light, these terms are preceded by the word "luminous." Other terms in general use with their definitions may be found in ASTM Method D 307[3] which should be consulted. In this connection it is important to distinguish the difference in meaning of the two terms "reflectance" and "reflectivity." The former refers to a property of an object or specimen, such as a coating-on-ground, where the indicated reflectance, total or directional, may be influenced by the reflectance of the substrate as well as that of the coating. On the other hand, reflectivity characterizes a material rather than a specimen and is determined at such a thickness of the material that any increase in thickness will not change the measured value.

In ASTM Method D 307[3] the symbol R_s designates luminous directional reflectivity. Total reflectance is usually designated by R and directional reflectance by R with subscripts indicating the angles of irradiation and reception respectively. For example, $R_{45,0}$ designates the directional reflectance of a specimen when irradiated at 45 deg and observed at 0 deg to the normal, and $R_{5,D}$ when irradiated at 5 deg and viewed diffusely. By the Helmholz reciprocal relation (**2**) it can be shown that these conditions of irradiation and observation can be interchanged without affecting the result, that is, $R_{45,0} = R_{0,45}$. Importance of specifying geometrical conditions becomes evident when it is known that the radiance of surfaces is usually a maximum in the group of directions near specular reflection and that this maximum becomes more pronounced as the angle of incidence increases (**3**).

REFLECTOMETRIC METHODS AND INSTRUMENTS

With the foregoing very brief and much-abridged review of pertinent fundamentals, let us now consider reflectometric methods and see how some of the more commonly available instruments fit into a significant reflectance test for curing materials. The two methods most widely used employ spectrophotometers and filter photometers respectively for making reflectance measurements. The former are capable of considerably higher precision than the latter and are usually used in referee tests. On the other hand, filter photometers provide means for making simple, rapid measurements, accurate enough for many purposes and at a reasonable cost.

In this section only five commercial instruments will be discussed in relation to the present problem. Those wishing to become acquainted with other instruments developed for this purpose should consult especially references (**2**,**4**, and **5**). The five instruments selected are all of the photoelectric type and will serve to illustrate the principles of application involved.

Spectrophotometry and Spectrophotometers:

Spectrophotometry consists in determining the ratio of two radiant energies, from sample specimen and standard specimen respectively, at successive wavelengths over the spectral range desired. By plotting this ratio (reflection factor) against wavelength, a complete spectrophotometric curve can be obtained, and the area under this curve represents the reflectance of the sample specimen relative to the standard specimen.

Since this procedure yields a ratio of two reflectances at each increment of wavelength, the resulting value of reflectance for the sample specimen is independent of the spectral distribution of the incident energy and the spectral response of the photocell. Hence it is equivalent to the result which would be obtained with a source having an equal energy spectrum, that is, one emitting the same amount of energy at all wavelengths, and a photocell having equal response at all wavelengths in the given range. The essential requirements are that the source have a continuous spectrum and the photocell a measurable response throughout the specified range.

If it is desired to know the reflectance of a specimen when irradiated by a source with known spectral energy distribution, the reflection factor at each increment of wavelength is multiplied by the relative energy of the source at that wavelength and the area under the resulting curve again integrated. This area must then be divided by the area under the curve obtained when the standard is similarly irradiated. Thus, reflectance measurements made by use of an incandescent lamp can be readily converted to reflectance values for irradiation by any other source whose spectral characteristics are known—for instance, average daylight (International Committee on Illumination (ICI), illuminant C) or any of the three sources whose energy distributions are given in Fig. 1.

Finally, in photometric problems involving appearance, the relative response of the observer's eye to visible radiation must be taken into account. The eye does not respond equally to equal amounts of radiation at all wavelengths. It is much more sensitive to radiation in the yellow-green region of the spectrum than in the blue or red and cannot perceive ultraviolet or infrared radiation at all. The relative brightness of equal amounts of energy is shown in the curve of Fig. 2 which is called the luminosity function and defines the response of the ICI standard observer (6). To evaluate the brightness of a specimen illuminated by a given source and viewed by the standard observer, the product of reflection factor of the specimen and relative energy of the source at each increment of wavelength must now be multiplied by the relative brightness at that wavelength and the area under the resulting curve integrated as before. Luminous reflectance of the specimen is then expressed as a ratio of the value so obtained to that for MgO when similarly illuminated and viewed. The calculation of luminous directional reflectance may be expressed as follows:

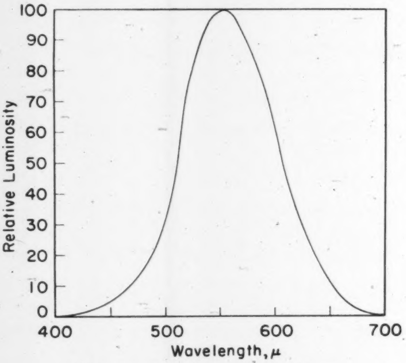

FIG. 2.—Luminosity Function.

$$R_{45,0} = \frac{\sum_{0}^{\infty} R_\lambda E_\lambda \bar{y}_\lambda \Delta\lambda}{\sum_{0}^{\infty} E_\lambda \bar{y}_\lambda \Delta\lambda} \quad \ldots\ldots\ldots (1)$$

where:

$R_{45,0}$ = luminous directional reflectance,

R_λ = spectral directional reflectance (reflection factor),

E_λ = spectral irradiance (relative energy of source),
$\bar{y}_\lambda$ = spectral luminosity (relative brightness), and
$\Delta\lambda$ = wavelength increment at which measurements are made.

Tables of values for E_λ, (H_λ, spectral irradiance in ASTM Method D 307) for the ICI standard illuminants A, B, and C, and for $\bar{y}_\lambda$ may be found in the literature (6).[3]

In general, spectrophotometers contain a lamp and a mechanico-optical system such that the radiant flux from the source is dispersed into its spectral components, which are then isolated successively in narrow bands to irradiate sample specimen and standard specimen. Reflected radiation is received by a photocell which measures the intensities of the reflected beams from the two surfaces. In the recording type of instrument, sample specimen and standard specimen are illuminated alternately in rapid succession and a continuous curve of comparative reflectance is drawn. In the manual type, sample specimen and standard specimen are alternately placed in the beam and the reflectance ratio is recorded for each increment of wavelength. Wavelength settings are also made manually. Examples of both types are described by Gibson (2).

The two recording spectrophotometers considered here are the latest model of the Hardy, or General Electric (GE), and the new Beckman DK-2R. Both employ integrating spheres for collecting the flux reflected from sample specimen and standard specimen, and the specular component can be either excluded from or included in the measurement. Illumination is at 6 deg in the GE instrument and 5 deg in the Beckman with diffuse reception in both. When the specular component is included, they give values of total reflectance, $R_{6,D}$ and $R_{5,D}$ respectively, but in any case cannot be used to measure luminous directional reflectance, $R_{45,0}$. An important distinction is the difference in range of the two instruments. As usually supplied, the GE covers an interval of about 0.38 to 0.80 μ while the Beckman has a range of 0.38 to 2.50 μ. The wide range of the latter makes it especially useful in the study of infrared reflectance.

The spectrophotometer used in the Michigan State Highway Department Research Laboratory is a Beckman DU with reflectance attachment and is of the nonrecording type. With a tungsten lamp it has a range of about 0.38 to 1.2 μ. This instrument does not have an integrating sphere, the specimen being illuminated perpendicularly and the reflected radiation collected at 45 deg by an ellipsoidal mirror ring which focuses it on the receiver. Thus the specular component is excluded from the measurement. There is a spread of about 20 deg about the axis of the reflected beam (35 to 55 deg) but the conditions closely approximate those recommended by the ICI for determining directional reflectance, $R_{45,0}$.

Filter Photometry and Photometers:

As we have just seen, the measurement of reflectance by spectrophotometric methods involves determining reflectance ratios at successive wavelengths in a given spectral range. With nonrecording instruments the determinations are made at such wavelength intervals that the spectrophotometric curve so obtained would not be changed significantly by taking further readings. Readings at 10-mμ intervals are usually adequate.

For some purposes, readings taken at several spectral regions of relatively narrow width may be sufficient. In abridged spectrophotometry, these regions are isolated by suitable optical filters selected for the particular application in mind.

While abridged spectrophotometry may have a limited application to reflectance measurements for curing materials, we are chiefly concerned here with filter photometry of another kind which is used in tristimulus colorimetry. In the latter method, three separate filters—amber, green and blue—of relatively wide spectral range are so selected that, in combination with a particular source and photocell, the tristimulus values defining the color of the sample under ICI standard conditions of illuminating and viewing may be directly determined (7). Without going into the subject of colorimetry, it is important to note that use of the green tristimulus filter in conjunction with a corresponding source and photocell may be made to yield a single value which is very nearly proportional to luminous reflectance. By comparison with a suitable standard, the reflectance of the specimen relative to that of MgO is easily found.

Probably the best known photoelectric reflectometers of the tristimulus type are the Hunter and the Photovolt. Both of these instruments are constructed so that the conditions of illuminating and viewing conform to ICI recommendations for determining directional reflectance, $R_{45,0}$. In the Hunter instrument, illumination is at 45 deg and viewing at 0 deg. In the Photovolt these conditions are reversed but equivalent. With the green tristimulus filter, both instruments give values of luminous directional reflectance relative to MgO from single readings on sample specimen and standard specimen, respectively. The specular component of reflected flux is excluded from measurements of $R_{45,0}$ in both instruments.

For the measurement of infrared reflectance, both instruments are designed to filter out visible radiation from the incident beam and to give reflectance values comparable to those obtained by the photographic method specified in Method 624.1 of Federal Specification TT-P-141b. In this method reflectance is defined in terms of the effect of the reflected energy on infrared-sensitive film when the specimen is irradiated by infrared energy from a 200-w tungsten lamp. It is pertinent to note that the response of the specified film does not extend beyond approximately 0.9 μ in the infrared.

SPECTRAL CHARACTERISTICS OF CURING MATERIALS

The third main factor entering into the design of a reflectance test for curing materials is the spectral characteristics of the materials themselves. To determine these characteristics, four types of material were selected for reflectance tests, namely, white curing paper, buff curing paper, white polyethylene sheets, and white membrane curing compounds. All materials met applicable ASTM requirements in other respects.

Reflectance Tests:

Samples of these materials were tested in four different ways: (1) 0-deg, 45-deg luminous directional reflectance by the Photovolt reflectometer; (2) 0-deg, 45-deg luminous directional reflectance by the Beckman DU spectrophotometer; (3) infrared reflectance by the Hunter and Photovolt reflectometers; and (4) 0-deg, 45-deg directional reflectance by the Beckman DU spectrophotometer over the wavelength range 0.31 to 1.20μ weighted by the spectral distribution of solar energy but not by the luminosity function. To reduce variables to a minimum for the purpose of comparing test methods, the polyethylenes and membrane compounds were tested for reflectivity rather than reflectance by using multiple thicknesses or coats.

Methods 1 and 2 cover the visible

TABLE II.—REFLECTANCE OF CURING MATERIALS.

Sample	Reflectance, per cent			
	Method No. 1[a]	Method No. 2[b]	Method No. 3[c]	Method No. 4[d]
WHITE CURING PAPER				
No. 1	57	58	64	60
No. 2	47	47	60	51
No. 3	49	50	56	51
No. 4	47	48	49	50
No. 5	42	42	45	43
No. 6	45	48	48	48
No. 7	54	55	62	57
No. 8	47	49	49	48
No. 9	44	47	45	46
BUFF CURING PAPER				
No. 10	32	32	44	38
No. 11	33	32	51	43
WHITE POLYETHYLENE				
No. 12	94	93	92	89
No. 13	92	92	91	89
No. 14[e]	77	76	79	73
WHITE MEMBRANE CURING COMPOUND				
No. 15	54	58	58	51
No. 16	80	82	84	76
No. 17	85	87	90	83
No. 18	76	77	84	74
No. 19	81	82	86	78
No. 20	81	83	85	78
No. 21	85	86	86	81
No. 22	84	86	86	80

[a] ASTM Method E 97 – 55, Photovolt reflectometer, NBS standard plaque 50Q reference.

[b] ASTM Method D 307 – 44, Beckman DU, MgO reference.

[c] TT-P-141b, Method 624.1. Hunter Multipurpose reflectometer used for white and buff papers, Photovolt reflectometer for white polyethylenes and membrane curing compounds.

[d] Modified ASTM Method D 307 – 44, Beckman DU, MgO reference, ordinates weighted by Moon's data for solar radiation in the range 0.3 to 1.2 μ

[e] Curing paper faced on one side with white polyethylene.

portion of the spectrum only and give values of reflectance which represent the "appearance" of the material when viewed by the ICI standard observer in average daylight (illuminant C). About 45 per cent of the total solar energy is included in this wavelength interval.

Method 3 includes in the measurement a wavelength range of about 0.7 to 0.9 μ, which contributes approximately 23 per cent of the total insolation. Two reflectometers were used in this method, the Hunter for the papers and the Photovolt for the polyethylenes and membrane compounds. This was necessary because of the lack of a complete set of secondary standards for either. Results from the two instruments checked to within 0.5 per cent in luminous directional reflectance determined on the same samples.

In method 4, the range of measurement includes more than 85 per cent of the total insolation. While this method does not include the specular component of reflected energy in the measurement, it unquestionably gives the most significant results of any of the four methods and was used as a standard for evaluation of the others.

Results and Discussion:

Data obtained from the four procedures applied to the four types of curing material are given in Table II and some illustrative spectrophotometric curves in Fig. 3. The curves in particular reveal some pertinent features of the different types of material.

1. The white papers are somewhat selective, with spectral reflectance reaching a broad maximum at around 0.7 to 0.8 μ, and declining very gradually or holding almost constant thereafter in the infrared. Sample No. 2 is a 2-yr-old paper of the same make as sample No. 1, and the depression of the curve in the visible region indicates the effect of age and possible bleeding of the bituminous layer through the surface. The three curves shown represent extremes

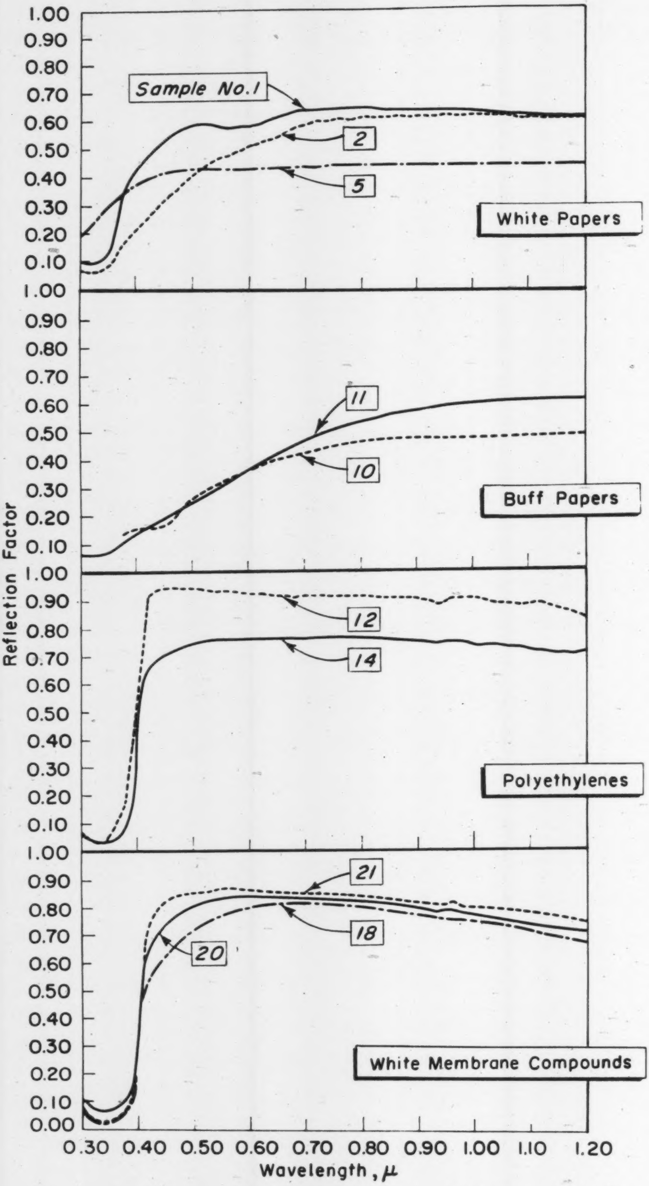

FIG. 3.—Spectral Reflectance of Curing Materials.

in characteristics of the nine samples of white paper tested, the others being intermediate in both spectral selectivity and directional reflectance. All of the curing papers were highly diffusing, having 60-deg gloss values of only 4 to 6 units, measured with a Photovolt Model 610 reflectometer and Carrara glass standard having a gloss of 96 units.

2. Spectrophotometric curves for both samples of buff paper show progressively decreasing reflectance from the red to the violet end of the visible spectrum. This comparatively low reflectance in the blue region gives rise to the characteristic brownish color of the kraft paper. Sample No. 11 is of special interest. Spectral reflectance of this sample continues to rise, although at a diminishing rate, past the visible region and out to a wavelength of about 1.1 μ in the infrared before levelling off. This property enables it to equal the least reflective "white" paper in reflectance determined in accordance with method No. 4 (Table II).

3. The two polyethylene films, samples Nos. 12 and 13, exhibited nearly identical characteristics, the spectral reflectivity rising almost vertically at 0.4 μ to a well-defined maximum at about 0.45 μ and then diminishing gradually with increasing wavelength through the visible and into the infrared regions. The curve for sample No. 14, curing paper faced with white polyethylene, shows the effect of film translucence on reflectance and spectral selectivity. A similar effect is produced by placing a white polyethylene sheet on a black background. All three samples exhibit appreciable specular reflection with 60-deg gloss values in the range of 35 to 50 units.

4. Finally, the spectrophotometric curves for white membranes are similar to those for the white polyethylenes in the given wavelength interval except for a falling off of spectral reflectance at the blue end of the visible region. Sample No. 18 had a distinct yellowish cast, with samples 20 and 21 showing progressive increase in whiteness. All but two of the samples produced relatively "flat" finishes with 60-deg gloss values of less than 20 units. The two exceptions, Nos. 18 and 19, gave gloss values of 86 and 40 units respectively, which suggests the possible desirability of limiting the gloss of membrane compounds used for highway construction in order to prevent objectionable glare on newly opened pavements.

RECOMMENDED TEST

As a final step in selecting a suitable reflectance test for curing materials, let us turn again to the data in Table II. Since existing preference is divided between the luminous reflectance method of ASTM Method E 97 and the infrared reflectance method of Federal Specification TT-P-141b, the primary comparisons will be of methods Nos. 1 and 3 respectively with method No. 4. Data for method No. 2 are included in the table only to indicate the degree of correlation that can ordinarily be expected between results from a spectrophotometer and filter photometer in a single test. As a matter of interest, spectrophotometric curves obtained by method No. 2 (ASTM Method D 307) gave nearly the same results for $R_{45,0}$ whether or not the ordinates were weighted by the luminosity function in the computations.

Reflectance values listed in Table II indicate that there is little to choose between methods No. 1 and No. 3 for measurements on white papers. Both gave reasonably close approximations to the more representative results obtained by method No. 4, although the values from method No. 1 were somewhat more consistent. Moreover, because the curing papers are highly diffusing, values for directional reflectance can be assumed to give a reliable indication of total reflectance.

For buff papers neither method No. 1 nor method No. 3 gave satisfactory results, the indicated value being too low in the former case and too high in the latter. These results can be deduced from the spectrophotometric curves of Fig. 3; the low reflectance in the visible region overinfluences the values obtained by method No. 1 and is not taken into account at all by method No. 3.

For the white polyethylene, both methods gave slightly higher values than method No. 4. Method No. 3 appears to have a little advantage for the plain white polyethylenes and method No. 1 for the polyethylene-faced paper. These conclusions could also be drawn from the spectrophotometric data given in Fig. 3.

While both methods produced reflectivity values for white membranes which were somewhat high, the results obtained by method No. 1 were consistently and appreciably nearer those obtained by the reference method and can be considered a satisfactory approximation. The spectrophotometric curves again provide the explanation: method No. 1 takes into account the moderate falling off of spectral reflectivity in the blue region, while method No. 3 does not.

SUMMARY

These tests indicate that ASTM Method E 97 is at least equal to Method 624.1 of Federal Specification TT-P-141b in significance and somewhat superior in consistency of results. In general, the former method yielded better reflection values for all of the white materials except the plain white polyethylene sheets. It should be emphasized, however, that both methods are empirical and depend for their validity on restriction to tests of relatively nonselective materials. We have already seen that neither method can be applied to the regular kraft papers or, by analogy, to other materials exhibiting highly selective spectral reflection.

On the basis of this study, then, it is concluded that ASTM Method E 97 for routine testing and ASTM Methods D 307 and D 791 for reference are the best that can be found at present using commercial photometric equipment currently available at a reasonable cost.

NOTE: It will be evident from the previous discussion that the only true indication of the ability of a given material to reflect solar energy would be furnished by an integrated measurement throughout all three regions of the solar spectrum, ultraviolet, visible and infrared. In such a measurement, total spectral reflection should be weighted by the energy distribution of solar radiation, but not by the luminosity function. Measurements of this kind can be made with sufficient accuracy by using the recording spectrophotometers described earlier. In most laboratories, however, the results would not justify the time and expense involved.

As a possible alternative it might be feasible to construct a reflectometer, or modify an existing one, so that an approximation of the true value of total reflectance could be obtained in a single reading. Following the design principles of the Hunter instrument (**7**), the source-filter-photocell conditions for such a measurement are that

$$E_s = E_I T s K \ldots\ldots\ldots\ldots (2)$$

at all wavelengths in the solar spectrum. In this equation E_s is the solar energy distribution, E_I the energy distribution of the source in the instrument, T the transmission of the filter, s the relative response of the receiver and K a constant. By using a thermopile as the receiver, s can be made equal to 1, and the problem reduces to one of finding a source-filter combination which will duplicate the energy distribution of solar radiation. No attempt has been made to find or make such a source-filter combination, and it is not known whether or not it is possible to do so.

Acknowledgment:

The study reported here was originally undertaken in connection with an assignment from C. E. Proudley, chairman of Subcommittee III-g for Methods of Testing and Specifications for Materials Used in Curing Concrete, of ASTM Committee C-9 on Concrete and Concrete Aggregates to Task Group VI of which W. W. McLaughlin, Testing and Research Engineer, was chairman. This assignment was to develop a recommended reflectance specification and method of test for white curing papers to include in ASTM Specification C 171[5] for curing paper. The work was performed under the general supervision of E. A. Finney, director of the Michigan State Highway Research Laboratory.

The authors also wish to acknowledge the painstaking work of O. W. Tompkins and R. D. Cook of the Spectroscopy and Photometry Section of the Michigan State Highway Research Laboratory who performed the necessary photometric tests and computations.

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[5] Specification for Waterproof Paper for Curing Concrete (C 171 – 56), 1958 Book of ASTM Standards, Part 4.

REFERENCES

(1) Parry Moon, "Proposed Standard Solar-Radiation Curves for Engineering Use," *Journal of the Franklin Institute*, Vol. 230, July–Dec., 1940, p. 583.

(2) K. S. Gibson, "Spectrophotometry," *Circular 484,* National Bureau of Standards, Sept. 15, 1949.

(3) J. W. T. Walsh, "Photometry," Constable and Co. Ltd., London (England). 2nd. Edition (1953).

(4) K. S. Gibson, "A Survey of Spectrophotometers," *Paper Trade Journal*, Technical Section, Vol. 111, p. 135 (1940)

(5) Committee on Colorimetry, Optical Society of America, "The Science of Color," Thomas Y. Crowell Co., New York, N. Y. (1953).

(6) A. C. Hardy, "Handbook of Colorimetry," Technology Press, Cambridge, Mass. (1936).

(7) R. S. Hunter, "Photoelectric Tristimulus Colorimetry with Three Filters," *Circular C 429*, National Bureau of Standards, July 30, 1942.

DISCUSSION

MESSRS. BRYANT MATHER[1] AND LEONARD PEPPER[1] (*presented in written form*).—The authors are to be complimented for making available the results of their significant evaluation of the factors and phenomena involved in reflectance testing of curing materials and the results of their comparison of available standardized methods of measuring reflectance. Our discussion is presented to raise one, probably minor, question concerning one conclusion and to describe the experience we have had with a specification limit on and test for reflectance of membrane-forming compounds for curing concrete.

The authors conclude that ASTM Method E 97[2] is at least equal to Method 624.1 of Federal Specification TT-P-141b in significance. This conclusion is certainly justified by the data the authors present and for the materials they tested. We would like to point out, however, that the ASTM method would not be as satisfactory for materials having the reflection characteristics of samples Nos. 12 and 21. The authors note that both methods "depend for their validity on restriction to tests of relatively nonselective materials." This is very important. The human observer can see and to some extent estimate the reflectance of a white material, but he cannot do the same for infrared reflectance. The ASTM method will give results that are more misleading than those that would be given by the Federal method in the case of the unusual material which has a rather sharp cut-off in reflectance in the infrared range. As is stated in the paper, the original objective of the authors' study was to recommend a specification and method of test for white curing papers. It, therefore, follows that, although liquid membrane-forming curing compounds were included in the study, the consideration of appropriate specifications and methods of testing them was not covered in detail.

The U. S. Army Corps of Engineers in 1952 modified its specifications for membrane-forming compounds for curing concrete to require that these materials be pigmented and that they be tested in a specified manner for reflectance. The test method adopted was based on that given in Methods 406.1 and 612.1 of Federal Specification TT-P-141b, involving the use of the Hunter multipurpose reflectometer used in the manner described in the National Bureau of Standards *Research Paper No. 1345* with a Wratten No. 88 filter. This procedure was selected because of previous successful experience with its use at the South Pacific Division Laboratory of the Corps of Engineers. The method has been used satisfactorily since 1952 by the three Corps of Engineers laboratories engaged in testing curing compounds for acceptance—the South Pacific Division Laboratory, the

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[1] Engineer and Chemist, respectively, Concrete Division, U. S. Army Engineer Waterways Experiment Station, Jackson, Miss.

[2] Method of Test for 45-deg, 0-deg Directional Reflectance of Opaque Specimens by Filter Photometry (E 97 – 55), 1958 Book of ASTM Standards, Parts 4, 5, 8, and 10.

Ohio River Division Laboratory, and the Concrete Division of the Waterways Experiment Station. The specification limit is not less than 65 per cent, and the test is performed on the surface of a mortar specimen. The specimen is a 1:3 graded Ottawa sand mortar of 0.53 water-cement ratio molded to the shape of a disk 8 in. in diameter, $\frac{3}{4}$ in. thick. The specimen is smooth trowelled and the compound is sprayed on at 200 sq ft per gal. The test is made after the specimen has been stored at 30 ± 3 per cent relative humidity; 100 ± 2 F, and in an atmosphere moving at 10 ± 3 mph over the specimen surface for not less than 24 hr. Obviously, most of these factors will affect the reflectance that is measured and hence need to be specified in order to develop a reproducible test and set appropriate specification limits.

The indications we have are that the reflectance test as specified by the Corps of Engineers is a test that has very good reproducibility. In the laboratory we generally obtain successive reflectance readings on a single test specimen that agree within ±1 per cent and only rarely do they show as much as ±2 per cent spread. The difference between successive readings is probably caused by differences in thickness of the membrane, differences in smoothness of the mortar surface, differences in moisture content of the mortar substrate, and similar factors. The good reproducibility of the test is also revealed by the fact that it is apparently possible for a manufacturer to produce a product with an average reflectance very close to the specified minimum and still have an acceptably high percentage of samples pass the test. The last 100 samples that were accepted from the production of a single manufacturer had reflectance test values distributed as follows:

Test Result	Number of Tests
70	1
69	13
68	9
67	19
66	25
65	33

It is also indicated by the progressive reduction in average reflectance of the product represented by these samples during the period covered. The average reflectance of the 100 samples was 66.5 per cent. The averages of consecutive groups of 10 samples were 66.9, 67.2, 66.9, 67.6, 66.9, 66.3, 65.5, 66.3, 65.4, and 65.7 per cent; or for the first 50, 67.1 per cent and for the second 50, 65.8 per cent.

MESSRS. H. JOHNS[3] AND P. W. LEWIS[3] (*by letter*).—The paper is interesting and quite comprehensive in the amount of testing done to determine a method for measuring the reflectance of curing materials. It is gratifying to note that the general conclusion arrived at, namely, that "the only true indication of a given material to reflect solar energy would be furnished by an integrated measurement throughout all three regions of the solar spectrum, ultraviolet, visible, and infrared," is similar to our thinking on the subject. However, there is some question in our minds whether it is necessary to consider all three bands since only a small amount of the energy contained in the ultraviolet band is converted into heat. While there is considerable energy in the ultraviolet band, most of it is dissipated in the surface of an object in the form of chemical energy. The significant bands are then, to our way of thinking, the visible and infrared bands so far as the consideration of heat absorption is concerned. Of these two bands, we believe the visible band to be more advantageous for curing compound tests.

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[3] Engineers, Division of Engineering Laboratories, Bureau of Reclamation, Denver, Colo.

Approximately 10 years ago, in our studies of white-pigmented compounds as a means of reducing concrete temperatures, we obtained good correlation between daylight reflectances and heat absorption in concrete as shown in the accompanying Fig. 4. It has been our experience that some white-pigmented compounds yellow considerably when exposed to weathering, with a resulting decrease in daylight reflectance. This, of course, increases the heat absorption, raising the temperature of the concrete mass.

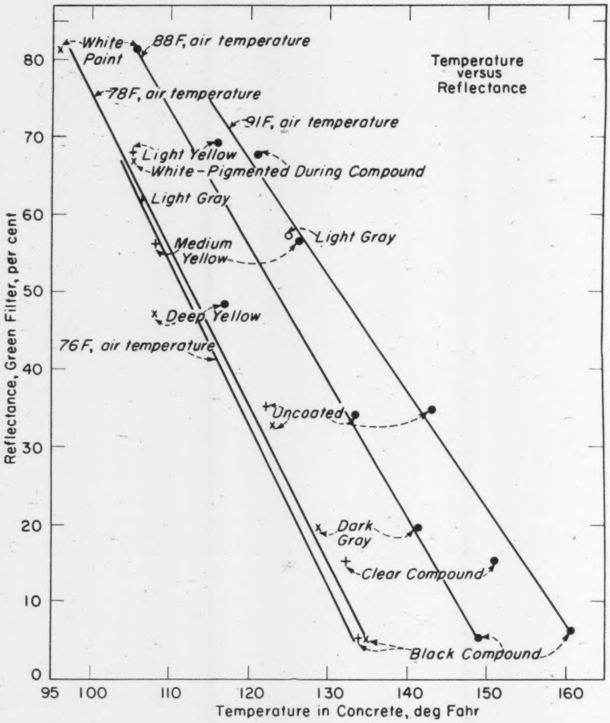

FIG. 4.—Correlation Between Daylight Reflectance and Heat Absorption in Concrete.

In recent brief tests on a set of specimens with varying degrees of yellowness, ranging from white to medium dark yellow, we found decreasing daylight reflectances as the yellowing increased, as expected, but little, if any, difference in relative infrared reflectance. Our exploratory data on this investigation, being brief, are not suitable for further presentation other than to mention it.

It is our belief that as yellowing occurs, the daylight reflectance decreases, resulting in increased absorption of the higher energy, shorter wavelength light, particularly in the blue region. There may or may not be a corresponding change in the infrared region.

In the subject paper, no mention is made of correlating surface and mass temperature with reflectance readings.

Since concrete temperature reduction is the principal reason for using white-pigmented sealing compounds, it seems that a study of the relationships of surface and mass temperatures with apparent daylight reflectance and infrared reflectance should be made before any particular method or combination is selected as a standard.

Based upon our knowledge and experience to date, however, if only one of the radiation bands is to be used in reflectance tests, the advantages appear to be in favor of the daylight reflectance test.

MESSRS. C. C. RHODES, M. H. JANSON, AND M. G. BROWN (*authors' closure*)—Bryant Mather and Leonard Pepper have presented some interesting information on the experience of the U. S. Army Corps of Engineers with reflectance tests of membrane-forming compounds for curing concrete. There are several statements in their discussion of the present paper, however, on which the authors would like to comment.

We cannot agree with the statement that "The ASTM method will give results that are more misleading than those that would be given by the Federal method in the case of the unusual material which has a rather sharp cut-off in reflectance in the infrared range." As pointed out in the paper, the applicable portion of the Federal method, which is essentially the same as Corps of Engineers method CRD-C 311-52, covers a wavelength range of approximately 0.7 to 0.9 μ in the infrared, which range includes only about 23 per cent of the total insolation. Can we say that reflection of this 23 per cent is more important or significant than reflection of the approximately 45 per cent of the total energy found in the visible range? Even if such an "unusual material" were found, a reflectance measurement by either the Federal method or the U. S. Army Corps of Engineers method could be even more misleading as an indication of total reflectance than one by ASTM Method E 97 because of the relative proportions of incident energy involved in the measurements. Actually, of all curing materials which we have tested spectrophotometrically so far, we have failed to find one of any type which is "white" in the visible range and "black" in the infrared.

In connection with specifications and tests of membrane curing compounds we should like to mention that, while the study reported in the present paper was initiated in connection with requirements for curing paper, Michigan has been specifying, testing and using white membrane curing compounds since 1949. Our first specification contained a requirement of a minimum daylight reflectance of 70 per cent relative to magnesium oxide and this requirement has been maintained in all subsequent issues. We should also like to call attention again to the reflectance values for white membrane compounds given in Table II, which indicate that results obtained by ASTM Method E 97 are consistently and appreciably nearer those obtained by the reference method than are those obtained by Federal Method 624.1.

The authors are in accord with the comments of Messrs. H. Johns and P. W. Lewis regarding the relative significance of ultraviolet incident energy in a reflectance test for concrete curing materials. As stated in the paper, energy in the ultraviolet range ordinarily constitutes only about 4 per cent, or less, of the total insolation. Although this amount is small and can be neglected in most cases, it should be included in a complete determination of total reflectance.

We concur also in the observation by Messrs. Johns and Lewis that white compounds sometimes yellow with exposure and that such yellowing would be accompanied by an increase in energy absorption. Because of the relatively short total exposure in a normal curing period and the fact that the first day or two is the most critical interval for stress control in the concrete, we do not believe that changes of this kind would be of major significance.

The data presented by Messrs. Johns and Lewis in Fig. 4 are timely and significant. They indicate that a change in temperature of 1 F corresponds roughly to a change in luminous directional reflectance of 2 per cent at that particular geographical location and confirm, qualitatively at least, results of similar experiments in other laboratories, including our own. In the matter of correlating surface and mass temperatures with reflectance values, we agree that a study of the relationship of temperature to luminous directional reflectance and infrared reflectance would be interesting, though difficult. There is some question in our minds that such a study would yield sufficient additional information to justify the delay it would cause in adoption of a standard reflectance test for concrete curing materials.

INFLUENCE OF VARIOUS FACTORS ON SULFATE RESISTANCE OF CONCRETES CONTAINING POZZOLAN*

By Milos Polivka[1] and Elwood H. Brown[2]

Synopsis

Concrete exposed to sulfate waters or alkali soils generally exhibits a rapid rate of deterioration because of the expansive forces developed during crystallization of sulfoaluminates.

Reported are results of a research program undertaken to determine the effects of several types of exposure and periods of curing on the sulfate resistance of concretes containing portland cements of high and very low C_3A content with and without pozzolan replacements. The three pozzolans employed with each one of the cements included calcined shale, fly ash, and pumicite. The two sulfate solutions used were a mixture of sodium and magnesium sulfates and a solution of sodium sulfate only. The effects of sulfate exposures were judged by several criteria: loss in weight, change in length, change in compressive strength, and visual observation.

From the results of this investigation it is concluded that the use of a high-quality pozzolan in concretes containing high C_3A cement will greatly improve their sulfate resistance; that the sulfate resistance of a concrete containing the plain cement of very low C_3A content is so high that no improvement through use of pozzolanic replacement is possible; that an extended period of curing beyond 28 days has little or no effect in improving sulfate resistance; that the sodium-magnesium sulfate attacks exposed concrete surfaces, causing surface scaling, whereas the sodium sulfate tends to penetrate into the concrete causing swelling and cracking without scaling of exposed surfaces; and that the loss-in-weight method seems to provide the most positive evaluation of potential sulfate resistance.

It has been well established by extensive investigations in the laboratory and by examinations of structures in the field that some portland-cement concretes tend to deteriorate and disintegrate if exposed to the continuous action of sulfate waters or to the action of alkali soils (**1**).[3] Also, it has been established that this physical disintegration occurs as the result of chemical reaction during which crystals of calcium sulfoaluminate are formed, producing expansive forces and causing disruption of the concrete (**2**). Laboratory investigations of the resistance

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* Presented at the Sixty-first Annual Meeting of the Society, June 22–27, 1958.

[1] Associate Professor of Civil Engineering, University of California, Berkeley, Calif.

[2] Associate Engineer, University of California, Berkeley, Calif.

[3] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 1098.

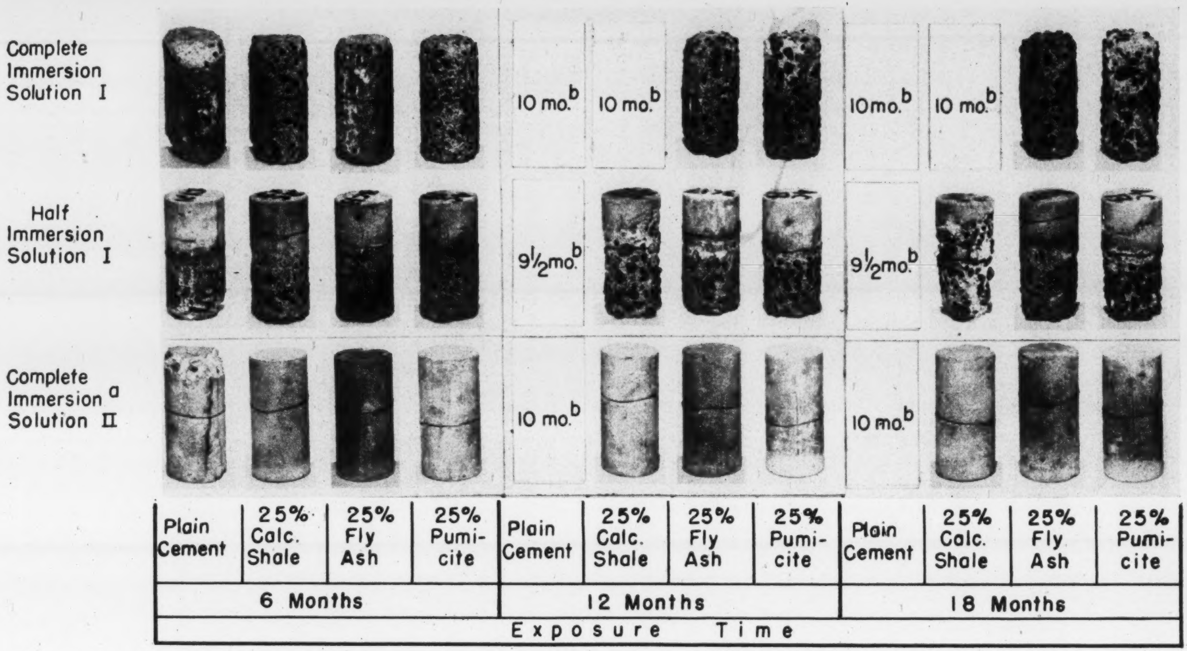
Complete Immersion Solution I
Half Immersion Solution I
Complete Immersion a Solution II
10 mo.b
10 mo.b
10 mo.b
10 mo.b
9½ mo.b
9½ mo.b
10 mo.b
10 mo.b
Plain Cement
25% Calc. Shale
25% Fly Ash
25% Pumi-cite
Plain Cement
25% Calc. Shale
25% Fly Ash
25% Pumi-cite
Plain Cement
25% Calc. Shale
25% Fly Ash
25% Pumi-cite
6 Months
12 Months
18 Months
Exposure Time

FIG. 1.—Effect of Period of Exposure on Concrete Containing High C_3A Cement (11.7 per cent) After 28-day Aging.

(*a*) Curing: Standard up to 14 days prior to sulfate exposure, when specimens were stored in air at 50 per cent relative humidity and 70 F.
(*b*) Failure: period of immersion to 25 per cent loss of weight.
Solution I: 5 per cent sodium sulfate plus 5 per cent magnesium sulfate;
Solution II: 2.1 per cent sodium sulfate.

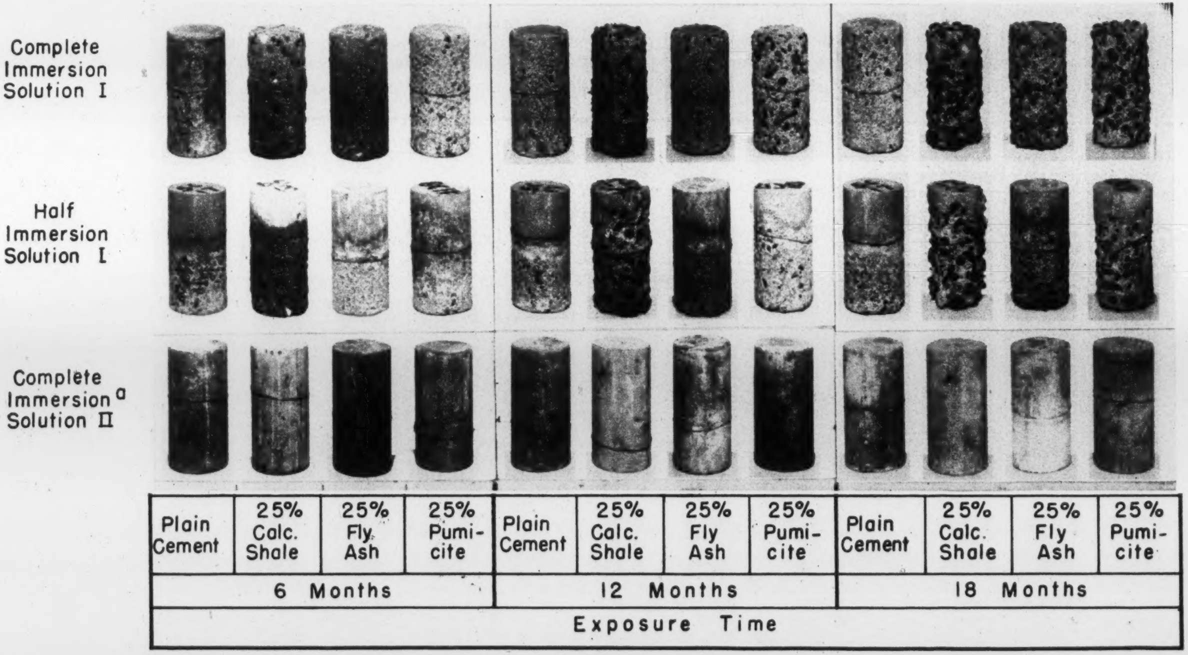
Complete Immersion Solution I
Half Immersion Solution I
Complete Immersion a Solution II
Plain Cement
25% Calc. Shale
25% Fly Ash
25% Pumi-cite
Plain Cement
25% Calc. Shale
25% Fly Ash
25% Pumi-cite
Plain Cement
25% Calc. Shale
25% Fly Ash
25% Pumi-cite
6 Months
12 Months
18 Months
Exposure Time

FIG. 2.—Effect of Period of Exposure on Concrete Containing Low C_3A Cement (2.8 per cent) After 28-day Aging.

(*a*) Curing: standard up to 14 days prior to sulfate exposure, when specimens were stored in air at 50 per cent relative humidity and 70 F.
Solution I: 5 per cent sodium sulfate plus 5 per cent magnesium sulfate;
Solution II: 2.1 per cent sodium sulfate.

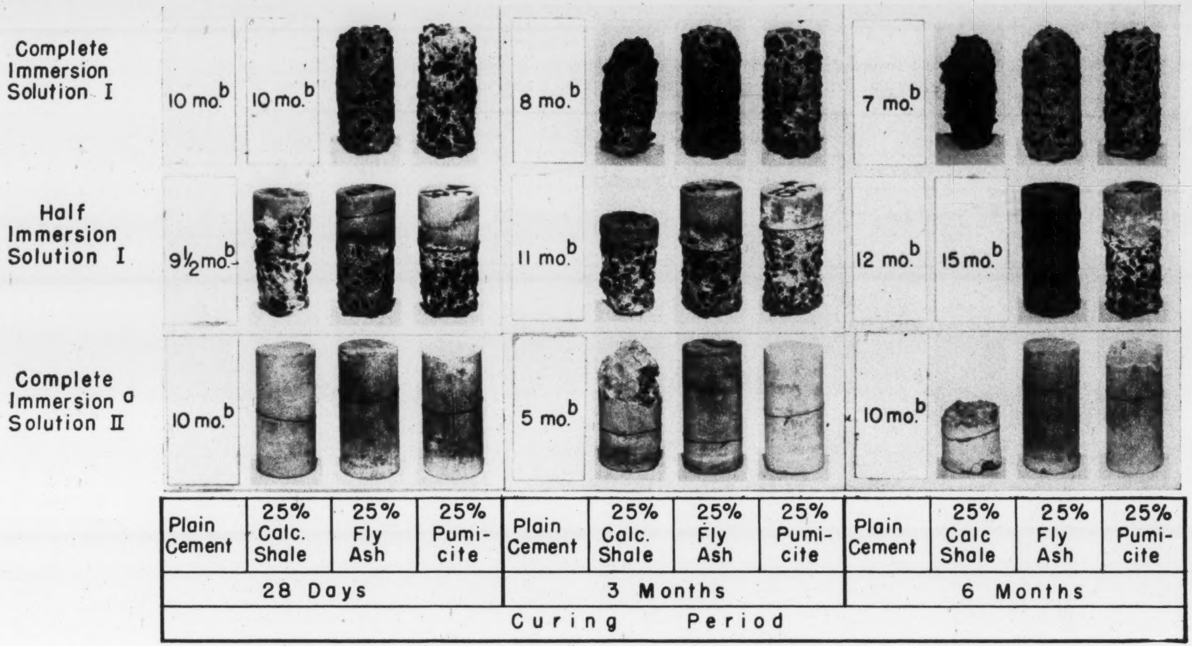
Complete Immersion Solution I
Half Immersion Solution I
Complete Immersion a Solution II
10 mo.b
10 mo.b
8 mo.b
7 mo.b
9½ mo.b
11 mo.b
12 mo.b
15 mo.b
10 mo.b
5 mo.b
10 mo.b
Plain Cement
25% Calc. Shale
25% Fly Ash
25% Pumi-cite
Plain Cement
25% Calc. Shale
25% Fly Ash
25% Pumi-cite
Plain Cement
25% Calc Shale
25% Fly Ash
25% Pumi-cite
28 Days
3 Months
6 Months
Curing Period

FIG. 3.—Effect of Period of Curing on Concrete Containing High C_3A Cement (11.7 per cent) After 18-month Exposure.

(*a*) Curing: standard up to 14 days prior to sulfate exposure, when specimens were stored in air at 50 per cent relative humidity and 70 F.
(*b*) Failure: period of immersion to 25 per cent loss of weight.
Solution I: 5 per cent sodium sulfate plus 5 per cent magnesium sulfate;
Solution II: 2.1 per cent sodium sulfate.

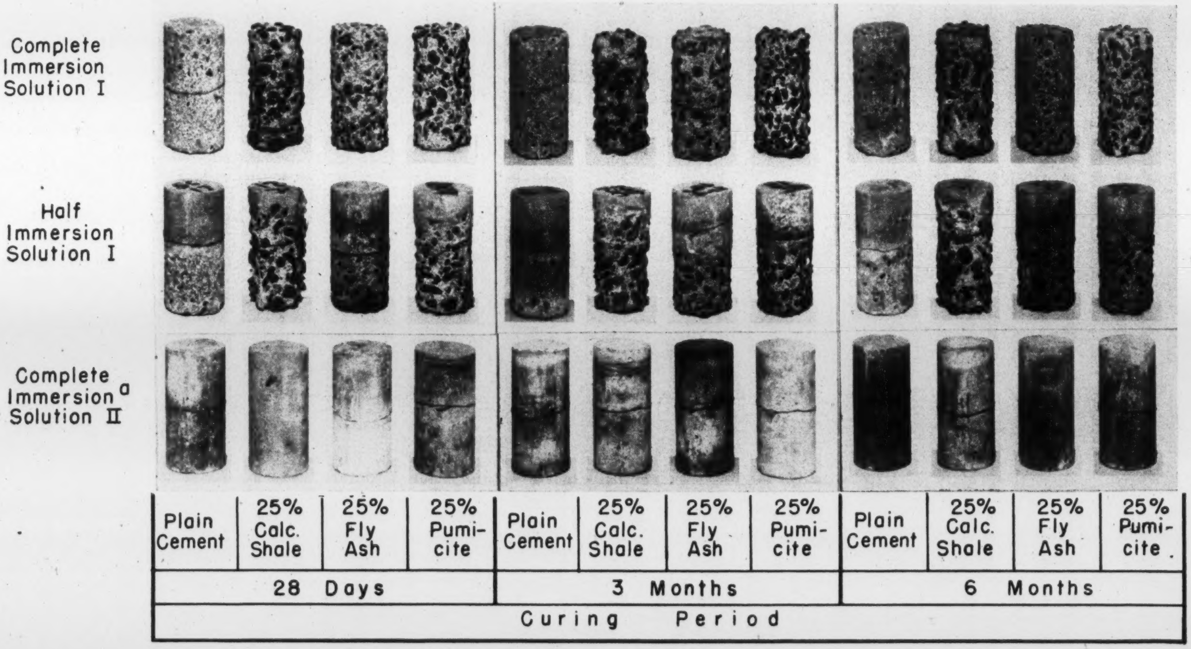
Complete Immersion Solution I
Half Immersion Solution I
Complete Immersion[a] Solution II
Plain Cement
25% Calc. Shale
25% Fly Ash
25% Pumicite
Plain Cement
25% Calc. Shale
25% Fly Ash
25% Pumicite
Plain Cement
25% Calc. Shale
25% Fly Ash
25% Pumicite
28 Days
3 Months
6 Months
Curing Period

FIG. 4.—Effect of Period of Curing on Concrete Containing Low C_3A Cement (2.8 per cent) After 18-month Exposure.

(*a*) Curing: standard up to 14 days prior to sulfate exposure, when specimens were stored in air at 50 per cent relative humidity and 70 F.
Solution I: 5 per cent sodium sulfate plus 5 per cent magnesium sulfate;
Solution II: 2.1 per cent sodium sulfate.

of portland-cement concretes have shown that the resistance to sulfate action is principally influenced by the tricalcium aluminate (C_3A) content of the portland cement (**3–7**). As the C_3A content increases above about 5 or 6 per cent, the resistance of the concrete to sulfate action decreases. ASTM specifications[4] for type V cement require the C_3A content not to exceed 5 per cent, the C_4AF plus twice the C_3A not to exceed 20 per cent, and the C_3S not to exceed 50 per cent.

Several research projects as well as field structures have demonstrated that the use of an active pozzolan of suitable quality as a replacement of a portion of the cement produces concrete that is considerably more resistant to sulfates than that produced by the portland cement alone. During the construction of the Colorado River Aqueduct for the Metropolitan Water District of Southern California, an extensive investigation of the sulfate resistance of both mortars and concretes containing portland cements and a variety of portland-pozzolan cement blends was undertaken at the District laboratory (**8**). The results of this investigation confirmed the conclusion that pozzolans of high quality used as partial replacements of medium to high C_3A-content portland cements substantially increased the resistance of concrete to sulfate action. Other investigations (**9–12**) made in the Engineering Materials Laboratory at the University of California have shown that pozzolans of suitable quality improve the sulfate resistance of concrete.

SCOPE OF PRESENT INVESTIGATIONS

The investigations described in this paper were undertaken to determine the effects of several types of exposure and the period of curing on the sulfate resistance of concretes containing portland cements of high and very low C_3A content with and without pozzolan replacements. The effects were judged by several criteria in order to determine the most suitable criterion for evaluating sulfate resistance.

In order to carry out this program and still keep the number of specimens and observations within practical limits, only three pozzolans were employed with each one of the two cements. Moist-curing periods were 14 days, 28 days, 3 months, and 6 months. After each particular period of curing, the specimens were immersed in each of two different sulfate solutions, namely, a mixture of sodium and magnesium sulfates, and a solution of sodium sulfate only. Corresponding control specimens were immersed in water.

TABLE I.—ANALYSES OF PORTLAND CEMENTS.

Constituent	High C_3A Portland Cement	Very Low C_3A Portland Cement
OXIDE COMPOSITION, PER CENT BY WEIGHT		
SiO_2	21.21	24.35
Al_2O_3	6.15	3.05
Fe_2O_3	2.70	3.11
CaO	63.32	65.94
MgO	2.83	1.06
SO_3	2.16	1.54
Loss on ignition	2.01	1.29
CALCULATED COMPOUND COMPOSITION, PER CENT BY WEIGHT		
C_3S	45.1	54.0
C_2S	26.9	29.2
C_3A	11.7	2.8
C_4AF	8.2	9.5
$CaSO_4$	2.2	1.6
$C_4AF + 2 \times C_3A$	31.6	15.1

For the sodium-magnesium-sulfate solution, the exposures were (*a*) complete immersion, (*b*) half immersion, and (*c*) daily cyclic treatment. For the sodium-sulfate solution, specimens were subjected to a 4-week cyclic wetting-and-drying treatment similar to that used by Higginson and Glantz (**7**). Four different methods of evaluating sulfate resistance were employed: (*a*) loss in weight, (*b*) change in length, (*c*) change in compressive strength, and (*d*) visual observation. A photographic record of the condition of the specimens during exposure to sulfates was also made at regular intervals. Samples of this photographic record are shown in Figs. 1 to 4.

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[4] Specifications for Portland Cement (C 150 – 56), 1958 Book of ASTM Standards, Part 4.

The investigation involved the casting, curing, and treatment of 2520 concrete specimens.

MATERIALS AND PREPARATION OF SPECIMENS

Cements:

Table I gives the chemical analyses of the two portland cements. The C_3A contents of the high and very low C_3A cements were 11.7 and 2.8 per cent respectively. The amount of C_4AF plus twice the C_3A was 31.6 per cent for the high C_3A cement and 15.1 per cent for the very low C_3A cement.

The very low C_3A cement was found in earlier investigations to have a very high sulfate resistance. It was selected to determine whether pozzolan replacements would produce concretes more resistant to sulfate action than those containing the very low C_3A cement without replacement.

Pozzolans:

The three pozzolans, each used in the amount of 25 per cent by weight of total cementing material (portland cement plus pozzolan), included a calcined opaline shale, a low-carbon fly ash, and a pumicite or volcanic ash. From previous investigations, each had been found to possess satisfactory pozzolanic activity and to produce concretes of very low permeability.

The values of specific gravity and fineness for each of the two cements and the three pozzolans are shown in Table II.

Aggregates:

The sand, principally quartz, was from Monterey, Calif. Its fineness modulus was 2.67.

The coarse aggregate was a waterborne gravel from the American River at Fair Oaks, Calif., ranging in size from passing No. 4 to ¾-in. sieve.

The aggregates were chosen for their excellent uniformity and quality. The individual particles of both sand and gravel were hard, sound, of low absorption, and highly resistant to sulfate action. The sand-aggregate ratio was 0.45 by weight.

Concretes:

The nominal cementing-material content of the concretes was 5 sacks (470 lb) per cu yd. The water content was adjusted to obtain a workability, as measured by the Powers' remolding effort, of 15 jigs. The consistency, as measured by slump, ranged from 3.0 to 3.7 in. Properties of fresh concretes are given in Table III. The data shown are average

TABLE II.—SPECIFIC GRAVITY AND FINENESS OF CEMENTING MATERIALS.

Cementing Material		Specific Gravity	Specific Surface,[a] sq cm per g
Portland cement	High C_3A (11.7 per cent)	3.14	3300
	Very low C_3A (2.8 per cent)	3.17	3500
Pozzolan	Shale, calcined at 1400 F	2.35	9050
	Fly ash, 1.8 per cent carbon	2.46	3600
	Pumicite, raw	2.33	4950

[a] Specific surface of portland cements determined by Blaine air-permeability method, and for pozzolans by Klein hydrometer.

values obtained from 15 batches for each of the eight concretes.

The water requirement of concretes containing calcined shale was about 19 per cent higher than that of the corresponding control mixes. For the concretes containing fly ash it was about 6 per cent lower, and for those containing pumicite about 3 per cent higher.

Test Specimens:

The specimens were 3 by 6-in. cylinders, three for each test condition, and were cast in steel molds. Each specimen to be used for determination of length change had a gage plug embedded in the center of each end.

TABLE III.—PROPERTIES OF FRESH CONCRETE.

Cementing material (cement or cement plus pozzolan) content, 5 sacks (470 lb) per cu yd
0–¾ in. maximum size aggregate, sand-aggregate ratio 0.45 by weight, fineness modulus of sand 2.67

Pozzolan	Water-Cement (Plus Pozzolan) Ratio, by weight	Powers' Remolding Effort, jigs	Slump, in.	Penetration of Kelly Ball, in.	Flow, per cent	Unit Weight, lb per cu ft	Air Content, per cent
HIGH C_3A CEMENT (11.7 PER CENT)							
None	0.53	14	3.4	1.8	109	152.1	2.6
Calcined shale	0.66	14	3.0	1.6	96	148.5	2.2
Fly ash	0.50	15	3.3	1.6	105	152.9	1.7
Pumicite	0.55	15	3.3	1.8	102	150.5	2.3
VERY LOW C_3A CEMENT (2.8 PER CENT)							
None	0.53	16	3.7	1.6	119	152.7	2.3
Calcined shale	0.65	14	3.1	1.4	103	149.0	1.9
Fly ash	0.49	14	3.5	1.4	106	153.5	1.6
Pumicite	0.55	15	3.5	1.5	99	150.9	2.0

Casting and Curing of Specimens:

The specimens were cast from batches of concrete mixed in a bowl-type tilting-drum mixer of 1-cu ft capacity. The mixing and casting were done in a room maintained at 70 F and 95 per cent relative humidity. Immediately after being cast, specimens were stored in fog at 70 F for a period of 24 hr. The molds were then removed and the concrete specimens to be immersed in the sodium-magnesium sulfate solution were cured in fog until the time of immersion. The specimens to be immersed in the sodium sulfate solution were cured in fog for 14 days, after which they were stored in air at 70 F and 50 per cent relative humidity until the time of immersion.

SULFATE EXPOSURES

Solutions:

The sodium-magnesium sulfate solution contained 5 per cent sodium sulfate plus 5 per cent magnesium sulfate. The plain sodium sulfate solution contained 2.1 per cent sodium sulfate.

For specimens completely immersed, the volume of solution was equal to the volume of the specimens. For specimens subjected to half immersion, the volume of sulfate solution was one half of the total specimen volume. In this latter case, the immersion containers were of a size which accommodated only the three specimens of a given condition. The lower half of each of the 3 by 6-in. concrete specimens was immersed in the sulfate solution, and the upper half of each was exposed to air at 70 F and 50 per cent relative humidity. A thin metal cover, perforated to fit over the three specimens, was used on each container to minimize evaporation of the solution.

At intervals of 28 days, while the specimens were removed from the solutions for observation, the containers were cleaned and filled with fresh solution.

Exposures:

In actual service, concretes are subjected to a variety of sulfate exposures, none of which can be exactly duplicated in the laboratory. Concrete pipe and drain tile, for example, would be subject to drying very infrequently. Footings, retaining walls, and slabs resting on the ground may have surfaces which are exposed to long periods of drying. Portions of piers, piles, and other structures in ocean water may be subjected to regular wetting-and-drying cycles due to tidal changes.

To simulate possible field exposures, a complete immersion, a half immersion, and a cyclic wetting-and-drying exposure was employed for concrete specimens subjected to the action of the sodium-magnesium sulfate solution. This cyclic treatment consisted of a daily cycle of 16 hr of immersion at 70 F, followed by 8 hr in circulating air at 130 F. For concrete specimens subjected to the action of the plain sodium sulfate solution, the cyclic treatment consisted of complete immersion for 27 days at 70 F, followed by one day of drying in air at 70 F and 50 per cent relative humidity. The solutions and the types of exposure are given in Table IV.

TABLE IV.—SULFATE SOLUTIONS AND TYPES OF EXPOSURE.

Solution of Immersion	Type of Exposure
Solution I: Mixed solution 5 per cent sodium sulfate plus 5 per cent magnesium sulfate	1. Complete immersion at 70 F 2. Half immersion at 70 F with upper half exposed to 50-per cent relative humidity 3. Cyclic treatment—Complete immersion at 70 F for 16 hr followed by drying in circulating air at 130 F for 8 hr. Five cycles a week and two days of complete immersion each week-end
Tap water (control)	Complete immersion at 70 F
Solution II: 2.1 per cent sodium sulfate	Complete immersion at 70 F for 27 days, followed by 1 day of drying at 70 F and 50-per cent relative humidity
Tap water (control)	Same treatment as that for the 2.1 per cent sodium-sulfate solution

METHODS OF EVALUATING SULFATE RESISTANCE

The following methods of evaluating the potential resistance of concretes to sulfate action were employed:

1. Disintegration as measured by loss in weight.
2. Change in length, as compared to length just prior to beginning of sulfate treatment.
3. Compressive strength as compared with that of corresponding control specimens of the same age immersed continuously in water, and
4. Progress of disintegration as judged by visual observation and from photographs of concrete specimens taken at regular intervals.

The following is a brief description of the procedure employed in connection with each method of evaluating potential resistance to sulfate attack.

Loss in Weight:

The initial weight of each of the three specimens of a test condition to be evaluated by the loss-in-weight method was determined just prior to beginning treatment in the sulfate solution. At intervals of 28 days, each specimen was subjected to a blast with an air jet at 80 psi for 1 min to remove surface water and loose grains; then it was weighed and its change in weight was computed. Values of loss in weight were plotted in a manner similar to the plots shown in Fig. 5. When a specimen exhibited 25 per cent loss in weight, it was considered to have failed.

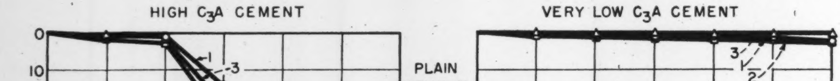

FIG. 5.—Effect of Sulfate Exposure on Loss in Weight of Concretes with Pozzolan. (28-day curing prior to sulfate exposure.)

1 = complete immersion } in 5 per cent sodium plus 5 per cent **magnesium sulfate.**
2 = half immersion }
3 = complete immersion in 2.1 per cent sodium sulfate.

Change in Length:

An initial reading of length was taken on each of the three specimens of a test condition prior to immersion in the particular sulfate solution. Specimens were observed for length at intervals of 28 days thereafter, and change in length was computed. The average value for the three specimens for each condition was plotted on a graph similar to Fig. 6. When the average expansion of the concrete specimens reached the value of 1500

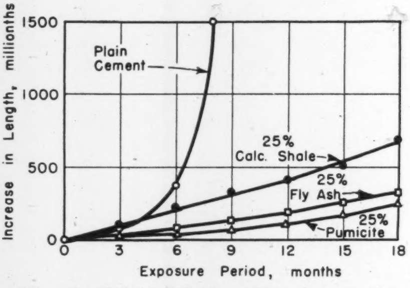

FIG. 6.—Effect of Sulfate Exposure on Expansion of High C_3A Cement-Concretes with Pozzolan.

Complete immersion (after curing for 28 days) in 5 per cent sodium plus 5 per cent magnesium sulfate.

TABLE V.—EFFECT OF PERIOD OF SULFATE EXPOSURE AND PERIOD OF CURING UPON COMPRESSIVE STRENGTH OF CONCRETES CONTAINING HIGH C_3A CEMENT AND POZZOLAN.

Pozzolan, 25 per cent by weight of cementing material	Solution[a]	Condition of Immersion	Compressive Strength of 3 by 6-in. Concrete Cylinders																	
			Cured 28 days						Cured 3 months						Cured 6 months					
			Exposed 3 months		Exposed 6 months		Exposed 12 months		Exposed 3 months		Exposed 6 months		Exposed 12 months		Exposed 3 months		Exposed 6 months		Exposed 12 months	
			psi	per cent	psi	per cent	psi	per cent	psi	per cent	psi	per cent	psi	per cent	psi	per cent	psi	per cent	psi	per cent
None, high C_3A cement	Water	Complete	5070	100	5280	100	5370	100	5240	100	5350	100	5300	100	5230	100	5280	100	5490	100
	I	Complete	4440	88	2640	50	[b]	[b]	4930	94	2990	56	[b]	[b]	4960	95	2650	50	[b]	[b]
	I	Half	4970	98	4200	79	[b]	[b]	5230	100	5210	97	[b]	[b]	5590	105	5620	106	[b]	[b]
	Water	Complete	4820	100	5080	100	5480	100	4880	100	4880	100	5090	100	4890	100	4950	100	5000	100
	II	Complete	4150	86	2260	45	[b]	[b]	3930	81	2970	61	[b]	[b]	4200	86	2600	53	[b]	[b]
Calcined shale	Water	Complete	4530	100	4800	100	4890	100	4700	100	4660	100	4950	100	4630	100	4560	100	5010	100
	I	Complete	4160	92	3900	81	3350	68	4530	96	3010	65	3340	67	3930	85	3530	77	2750	55
	I	Half	4050	89	3940	82	3320	68	4410	94	4050	87	3260	66	4430	96	4240	93	2670	53
	Water	Complete	4360	100	4410	100	4860	100	4630	100	4790	100	5200	100	4670	100	5120	100	4790	100
	II	Complete	4640	106	4670	105	4470	92	4720	102	4000	84	2600	50	4090	88	3440	67	2810	59
Fly ash	Water	Complete	5610	100	6090	100	6390	100	6060	100	6030	100	6430	100	6100	100	6360	100	6380	100
	I	Complete	5700	102	5590	92	4380	68	6180	102	5530	92	4600	71	5810	95	5570	88	4540	71
	I	Half	5530	99	6190	102	5930	93	6270	105	6410	106	5750	89	6080	100	6170	97	5410	85
	Water	Complete	5480	100	5900	100	5940	100	5640	100	5910	100	6260	100	6160	100	6280	100	6180	100
	II	Complete	5430	99	5700	95	5460	92	5820	103	5980	101	5980	96	6060	98	6400	102	6410	103
Pumicite	Water	Complete	5220	100	5840	100	6190	100	5580	100	5970	100	6280	100	6050	100	6120	100	6240	100
	I	Complete	5350	103	4870	83	4330	70	5220	94	5260	88	4020	64	5890	96	5470	89	3900	62
	I	Half	4990	96	5110	87	5260	85	5760	103	5670	95	5640	90	6090	101	6290	103	5720	92
	Water	Complete	5410	100	5770	100	6320	100	5500	100	5900	100	6650	100	6070	100	6280	100	6530	100
	II	Complete	5210	96	5750	100	6180	98	5560	101	5610	95	5770	87	5300	87	5320	85	4370	67

[a] Sulfate solutions: I = 5 per cent sodium sulfate plus 5 per cent magnesium sulfate.
II = 2.1 per cent sodium sulfate.

[b] Cylinders had disintegrated to such an extent that a satisfactory test could not be made.

TABLE VI.—EFFECT OF PERIOD OF SULFATE EXPOSURE AND PERIOD OF CURING UPON COMPRESSIVE STRENGTH OF CONCRETES CONTAINING VERY LOW C_3A CEMENT AND POZZOLAN.

Pozzolan, 25 per cent weight of cementing material	Solution[a]	Condition of Immersion	Compressive Strength of 3 by 6-in. Concrete Cylinders																	
			Cured 28 days						Cured 3 months						Cured 6 months					
			Exposed 3 months		Exposed 6 months		Exposed 12 months		Exposed 3 months		Exposed 6 months		Exposed 12 months		Exposed 3 months		Exposed 6 months		Exposed 12 months	
			psi	per cent	psi	per cent	psi	per cent	psi	per cent	psi	per cent	psi	per cent	psi	per cent	psi	per cent	psi	per cent
None, very low C_3A cement	Water	Complete	5680	100	6040	100	6300	100	6150	100	6100	100	6420	100	6150	100	6220	100	6360	100
	I	Complete	5880	103	6070	100	6060	96	5960	97	6610	108	6160	96	6600	107	6340	102	6400	101
	I	Half	5840	103	6380	106	6160	98	6210	101	6550	107	6490	101	6960	113	6790	109	6940	109
	Water	Complete	5910	100	5960	100	6520	100	6170	100	6250	100	6470	100	6180	100	6460	100	6550	100
	II	Complete	6160	104	6650	111	7110	109	6170	100	6290	101	6760	104	6250	101	6340	98	6610	101
Calcined shale	Water	Complete	4790	100	5140	100	5730	100	5190	100	5440	100	5580	100	5370	100	5600	100	5650	100
	I	Complete	4210	88	4110	81	3650	64	3840	74	4510	83	3570	64	4500	84	4620	82	3920	69
	I	Half	4470	93	4300	85	3930	68	5080	98	4780	88	4530	81	4880	91	4610	82	4670	83
	Water	Complete	4730	100	5190	100	5680	100	5180	100	5610	100	5730	100	5450	100	5680	100	5770	100
	II	Complete	4840	102	5130	99	5400	95	5140	99	5460	97	5760	101	5280	97	5800	102	5520	97
Fly ash	Water	Complete	6080	100	6910	100	7460	100	6770	100	7310	100	7540	100	7330	100	7380	100	7950	100
	I	Complete	6410	105	6610	96	6400	86	6760	100	6940	95	6170	82	7270	99	6910	94	6280	77
	I	Half	5490	90	6100	88	6590	88	6750	100	6960	95	7010	93	7310	100	7640	103	7230	89
	Water	Complete	5920	100	6710	100	7430	100	6960	100	7040	100	7340	100	7090	100	7210	100	7960	100
	II	Complete	6190	105	6820	102	7270	98	6820	98	7150	102	7390	101	7180	101	7370	102	7570	95
Pumicite	Water	Complete	5630	100	6180	100	6790	100	6200	100	6580	100	6750	100	6430	100	6890	100	7010	100
	I	Complete	5410	96	5210	84	4150	61	5510	89	4950	75	5310	79	5680	88	5160	75	5390	77
	I	Half	5440	97	5810	94	5690	84	5910	95	6380	97	6190	92	6610	103	6460	94	6330	90
	Water	Complete	5740	100	6390	100	7000	100	6450	100	7020	100	7240	100	6690	100	6960	100	7350	100
	II	Complete	5530	96	6130	96	6610	94	6350	98	6620	94	6820	94	6630	99	6890	99	6950	95

[a] Sulfate solutions: I = 5 per cent sodium sulfate plus 5 per cent magnesium sulfate.
II = 2.1 per cent sodium sulfate.

millionths (0.15 per cent), failure was considered to have occurred.

Change in Compressive Strength:

At the conclusion of specified periods of exposure, namely, 3, 6, and 12 months, three specimens of each test condition, if not too badly disintegrated, were tested for compressive strength. Also tested were the companion control specimens immersed in water. Tables V and VI give the average values of compressive strength for all test conditions. The change in compressive strength was computed as the difference in strength of specimens immersed in sulfate solution and the strength of corresponding specimens immersed in water. This difference, expressed in per cent, is also given in each table. Compressive strengths were computed on the basis of original cross-sections.

Visual Observation:

At intervals of 28 days, after the start of treatment, each loss-in-weight specimen was inspected, and at intervals of 3 months the one specimen selected to be representative of the average of the three specimens of a test condition was photographed in order to have a visual record of the progress of disintegration. These photographs were mounted on sheets of cardboard. Each sheet represented the set of photographs taken for a particular curing period (28 days, 3 months, or 6 months), and for exposure periods of 3, 6, 9, 12, and 18 months. Each type of portland cement was represented by fifteen of these sheets. Figures 1 to 4 show selected groups of photographs which were reduced in scale from the full-size sheets.

TEST RESULTS

The results of this investigation are discussed under the following headings: (1) Effect of type and period of sulfate exposure; (2) Effect of length of curing period prior to sulfate exposure; (3) Types and degree of disintegration caused by various sulfate exposures; and (4) Evaluation of methods of test for sulfate resistance.

Effect of Type and Period of Sulfate Exposure:

The progress of disintegration with time of exposure (6, 12, and 18 months) to sulfate attack of concretes cured for 28 days is illustrated in Figs. 1 and 2. Concretes shown in Fig. 1 contained the high C_3A (11.7 per cent) cement and those in Fig. 2 contained the very low C_3A (2.8 per cent) cement. In the first (left-hand) row in each set of photographs are specimens containing plain portland cement; in rows two, three, and four are specimens in which 25 per cent by weight of the portland cement was replaced by calcined shale, fly ash, or pumicite respectively.

High C_3A Cement—Sodium-Magnesium-Sulfate Exposure.—A study of Fig. 1 shows that concretes containing the plain cement and immersed in the sodium-magnesium-sulfate solution (5 per cent sodium sulfate plus 5 per cent magnesium sulfate) disintegrated rapidly regardless of whether completely or half immersed. In both cases the specimens had disintegrated completely after only 10 months of exposure.

Figure 1 shows also that a 25 per cent replacement of the plain portland cement by either fly ash or pumicite resulted in concretes of superior sulfate resistance as compared with the plain portland cement. Exposure to either complete or half immersion for 18 months was required before these specimens exhibited considerable surface scaling and spalling, exposing the larger pieces of aggregate.

In contrast to the high degree of resistance to sulfate attack exhibited by concretes containing fly ash and pumicite, those containing calcined shale, when subjected to complete immersion, failed after 10 months of exposure. The specimens subjected to half immersion were near failure after 18 months of exposure. A possible cause for the poor performance of concretes containing this calcined shale might be their higher water requirement (19 per cent higher than that of the corresponding plain cement concrete). A possible explanation of the poor sulfate resistance of concretes containing calcined shale was the fact that the shale was calcined at only about 1400 F. In previous investigations (9,10) the same type of shale, calcined at 1600 F showed a great improvement in sulfate resistance compared with that obtained for concrete containing plain portland cement.

The loss-in-weight data for concretes cured 28 days and containing the high C_3A cement are given in the left half of Fig. 5, for both complete and half immersion. These data again show the rapid disintegration of concretes containing the plain high C_3A cement, failure occurring after 10 months of exposure. The completely immersed concrete containing calcined shale failed also after 10 months of exposure, while the concretes containing fly ash or pumicite were only approaching failure after 18 months of exposure—pumicite concrete failing somewhat more rapidly than fly ash concrete.

Examination of Fig. 6, which was prepared from change-in-length measurements of a different group of specimens from those used for loss in weight and photographic record, shows the same general trend of degree of disintegration as the curves of Fig. 5. This is the case for both complete and half immersion. As judged by the change-in-length criterion, specimens containing the plain portland cement expanded very rapidly and failed after only 8 months of exposure. Although specimens containing calcined shale expanded more slowly than those containing plain portland cement, their expansion was considerably greater after 18 months of exposure than that of specimens containing either fly ash or pumicite. The low expansion of concretes containing fly ash or pumicite—and even the expansion of those containing calcined shale—indicates very markedly the improvement in the sulfate resistance of concrete through the use of pozzolan as a replacement of the high C_3A cement.

Table V gives the average compressive strengths of concretes exposed to sulfate action for periods of 3, 6, and 12 months. These results were obtained on a set of specimens separate from those used for either the loss-in-weight or the change-in-length tests. In the column next to each strength value the percentage of this strength is shown as compared to the strength of control specimens cured continuously in water at 70 F and tested at the same age as the exposed specimens. Examination of Table V shows that specimens subjected to either complete or half immersion indicate the same trends regarding the effectiveness of pozzolans in resisting sulfate attack as those noted from Figs. 5 and 6 for loss in weight and change in length. Prior to the end of the 12-month period of exposure, concretes containing the plain high C_3A cement disintegrated to such an extent that a satisfactory compressive strength test could not be made. After 12 months of exposure, the specimens containing fly ash and pumicite had lost only 20 to 30 per cent of their potential strength. These compressive strength results again indicate the superior resistance to sodium-magnesium-sulfate attack of concretes containing pozzolan as a replacement of the high C_3A cement.

High C_3A Cement—Sodium Sulfate Exposure.—Figure 1 shows that concretes containing the plain portland cement disintegrated as rapidly in the 2.1 per cent sodium sulfate solution as they did in the sodium-magnesium sulfate solution. Again the specimens failed after only 10 months of treatment. However, concretes containing any one of the three pozzolans exhibited practically no distress after 18 months of complete immersion in the sodium sulfate solution. The appearance of these specimens again illustrates the superior resistance to sulfate attack of concretes in which high C_3A cement is partially replaced by pozzolan.

Loss-in-weight data (left half of Fig. 5) also indicate the high resistance to sulfate action of concretes containing pozzolan. In this figure, the curves labeled 3 are for the specimens subjected to complete immersion in the 2.1 per cent sodium sulfate solution. The curve for concretes containing the plain portland cement shows that the specimens failed after 10 months of exposure, while those for concretes containing calcined shale, fly ash, or pumicite showed no appreciable loss in weight up to 18 months of exposure.

Curves similar to those of Fig. 6 were prepared to show the change in length of concrete specimens exposed to sodium sulfate solution. As judged by the change-in-length criterion (0.15 per cent expansion), even in this weak solution concretes containing the plain portland cement expanded very rapidly and failed after only 7 months of exposure. On the other hand, after 18 months of exposure specimens containing pozzolan showed expansions as follows: calcined shale, 530 millionths; fly ash, 280 millionths; and pumicite, 260 millionths. Such expansions are very small as compared with that of the plain portland cement concrete.

From Table V it is seen that the compressive strengths of concretes indicate approximately the same trends regarding the effectiveness of pozzolans in resisting the attack of this sodium sulfate solution as was noted from Figs. 5 and 6 for loss in weight and change in length. Again, as in the case of exposure in the sodium-magnesium sulfate solution, prior to the end of the 12-month period of exposure concretes containing the plain high C_3A cement disintegrated to such an extent that a satisfactory compressive-strength test could not be made, whereas those containing calcined shale, fly ash, or pumicite showed compressive strengths only about 10 per cent lower than those of control specimens immersed in water. These results indicate the superior resistance to sodium sulfate attack of concretes containing pozzolan as a replacement of high C_3A cement.

Low C_3A Cement—Sodium-Magnesium Sulfate Exposure.—Figure 2 shows photographs of concrete specimens containing the very low C_3A (2.8 per cent) cement. These photographs show that the plain portland-cement concrete exhibits high resistance to sulfate attack, whether subjected to complete immersion or to half immersion in the sodium-magnesium sulfate solution. After 18 months of exposure, only a very thin layer had scaled from the surface of each specimen. In contrast to the behavior of the concrete containing this plain cement, the concretes containing the pozzalans exhibited a greater degree of surface scaling. The concrete containing the calcined shale showed the most severe deterioration while that containing the fly ash showed the least.

The loss-in-weight data of concretes either completely or half immersed in this solution are shown in the right half of Fig. 5. After 18 months of complete immersion, the loss in weight for the plain very low C_3A cement concrete was only about 2 per cent, whereas for concrete containing the calcined shale it was 24 per cent; fly ash, 10 per cent; and pumicite, 18 per cent. The loss in weight was somewhat less for specimens only half immersed after the same period of exposure. These results show that concretes containing the very low C_3A cement were highly resistant to sulfate attack. The resistance of concrete containing plain low C_3A cement was superior to that of concretes containing pozzolan replacements.

After 18 months of complete immersion in the sodium-magnesium sulfate solution, plain low C_3A cement concrete as well as concretes containing fly ash or pumicite expanded only about 250 millionths, and concrete containing calcined shale expanded 320 millionths. All specimens which were half-immersed showed no measurable expansion.

Compressive strengths of concretes containing this very low C_3A cement are shown in Table VI. For a curing period of 28 days and for specimens containing the plain cement, the loss in strength after 12 months of either complete or half immersion was less than 5 per cent of the strength of the control specimens. In contrast, after 12 months of exposure the loss in strength for concretes containing fly ash was 15 per cent; for calcined shale about 35 per cent; and for pumicite 39 per cent for complete immersion and 16 per cent for half immersion.

From the results of these tests and observations, it is obvious that the resistance to sodium-magnesium sulfate attack of concrete containing the plain portland cement of very low C_3A content (2.8 per cent) was so high that no improvement through the use of pozzalanic replacements was possible. Actually, use of pozzolan replacement reduced somewhat the sulfate resistance of low C_3A cement concretes.

Low C_3A Cement—Sodium-Sulfate Exposure.—From the appearance of the specimens shown in Fig. 2, it would seem that very little or no disintegration has taken place even after an exposure of 18 months to sodium sulfate attack. From this visual examination, it could not be said that any one cement was better than another in resisting sulfate attack up to 18 months of exposure.

Also, the right half of Fig. 5 shows that for each of the four diagrams the loss-in-weight curve 3 is almost superimposed on the abscissa line of the diagram. The maximum loss in weight was in no case more than 1 per cent after 18 months of exposure.

The change-in-length data show that for each of the concretes the expansion was small. The value for the plain portland cement after 18 months of exposure was 340 millionths and for each of the specimens containing pozzolan the value was less than 250 millionths.

Table VI shows that, after 12 months of exposure, the compressive strength of plain-cement-concrete specimens had increased 9 per cent and that of the specimens containing the pozzolans had decreased not more than 6 per cent of that of their respective control specimens.

For this 2.1 per cent sodium sulfate solution, all of these results confirm the high resistance to sulfate attack of concretes containing this very low C_3A cement without pozzolan replacement. It is to be noted, however, that concretes containing pozzolan appear to have almost equal resistance to sodium sulfate attack.

Effect of Period of Curing:

The effect of period of curing on the disintegration of concrete exposed to sulfate attack is shown in Figs. 3 and 4. The photographs of these figures show the condition of specimens after 18 months of exposure.

High C_3A Cement—Sodium-Magnesium Sulfate Exposure.—It is seen from Fig. 3 that there are only minor differences between the specimens cured for 3 or 6 months and those cured for 28 days. The longer periods of curing seem to be slightly beneficial to the completely immersed calcined shale specimens but detrimental to those which were only half immersed. For the plain portland-cement concrete the reverse seems to be true, that is, the longer periods of curing reduce the resistance of the completely immersed specimens and increase slightly the resistance of the half-immersed specimens.

The loss-in-weight data up to 18 months of exposure, for specimens either completely or half immersed, after 3 or 6 months of curing paralleled quite closely the data shown for concretes cured for 28 days (Fig. 5). The loss in weight of specimens containing fly ash and pumicite was almost identical for all three curing periods, and only slight variations were observed for concretes containing plain portland cement or calcined shale.

Changes in length up to 18 months of exposure for specimens either completely or half immersed, after 3 or 6 months of curing, paralleled very closely those for the 28-day curing period (Fig. 6); the extended curing period slightly improved the resistance of the plain portland-cement concrete, but had little or no effect on concretes containing pozzolan.

Compressive strength values given in Table V indicate that the longer periods of curing (3 and 6 months) are somewhat beneficial to the plain-cement concrete subjected to half immersion; for all other concretes exposed to the sodium-magnesium sulfate solution, the period of curing appears to have no marked effect.

These results indicate that a long period of curing, up to 6 months, has little or no effect on the resistance of concretes containing high C_3A cement, with or without pozzolan, to the action of sodium-magnesium sulfate.

High C_3A Cement—Sodium Sulfate Exposure.—Figure 3 does not reveal any definite effect of period of curing upon specimens containing the plain portland cement. For the specimens containing the calcined shale, however, the longer period of curing appears to have a detrimental effect upon resistance to the action of the solution. There is some indication, also, that specimens containing the other two pozzolans, particularly the pumicite, suffer a slightly adverse effect from the longer period of curing.

Loss in weight of specimens after 3 or 6 months of curing, except for the calcined shale, agrees closely with that of concretes shown in Fig. 5 for 28-day curing. However, for the specimens containing the shale the loss in weight after 18 months of exposure was negligible for the 28-day curing period, 16 per cent for the 3-month curing period, and 25 per cent for the 6-month curing period.

Changes in length of specimens up to 18 months, after 3 or 6 months of curing, paralleled those shown in Fig. 6 for 28-day curing. For the plain cement, the change in length was almost identical for all three curing periods. Concretes containing calcined shale and cured for 3 months failed after 13 months of exposure, and those cured for 6 months failed after only 8 months of exposure. The expansion up to 18 months of exposure, of concretes containing fly ash or pumicite after 3 and 6 months of curing was about double that of concretes cured for 28 days.

The compressive strength values of Table V indicate that a long curing period (up to 6 months) has a decidedly detrimental effect on specimens containing calcined shale when exposed to sodium sulfate. Longer periods of curing also seem to have some detrimental effect on specimens containing pumicite. The specimens containing fly ash, however, seem to show a definite increase in strength and beneficial effects from the longer curing periods.

For concretes containing high C_3A cement and exposed to sodium sulfate, the extended period of curing for those containing calcined shale is extremely deleterious, for those containing fly ash or pumicite it is slightly deleterious, and for those containing the plain portland cement it has no effect.

Low C_3A Cement—Sodium-Magnesium Sulfate Exposure.—From Fig. 4 it would be difficult to say that the longer periods of curing, 3 and 6 months, had either a beneficial or a detrimental effect upon the concretes containing the low C_3A cement either plain or with pozzolan.

Loss-in-weight data also show no beneficial or detrimental effect from the longer period of curing.

Change-in-length data again indicate that the extended curing period has little or no effect on expansion of concretes.

Table VI shows that the extended curing period is somewhat beneficial to compressive strength of concretes containing plain low C_3A cement exposed to sodium-magnesium sulfate, whereas it has practically no effect on concretes containing pozzolan.

These results indicate that the extended period of curing has little or no effect on the resistance of concretes containing low C_3A cement, with or without pozzolan, when exposed to the action of sodium-magnesium sulfate.

Low C_3A Cement—Sodium Sulfate Exposure.—From Fig. 4 it appears that all of the specimens, regardless of curing period, are in perfect condition.

None of the concretes containing the low C_3A cement exposed to sodium sulfate showed any loss in weight, regardless of curing period.

Change-in-length data show that expansions of concretes were not affected by the extended curing period. Although loss-in-weight data and visual inspection indicated all concretes to be in perfect condition, the greatest expansion after 18 months of exposure occurred for the plain portland cement (300 millionths). This larger expansion of the plain portland-cement concrete perhaps indicates somewhat more rapid and thorough penetration of the sodium sulfate solution into these specimens than into the specimens containing the pozzolan replacements.

Compressive strengths of concretes containing pozzolan (Table VI) and exposed to sodium sulfate attack were approximately the same for all periods of curing as that of their respective control specimens. For plain portland-cement concrete, however, the extended curing period appeared to have a slightly detrimental effect.

These results clearly indicate that the period of curing of concretes containing low C_3A cement had no marked effect upon their resistance to sodium sulfate attack.

Effect of Type of Sulfate Exposure:

Sodium-Magnesium Sulfate.—The effects of type and degree of disintegration for complete and half immersions are shown in Figs. 1 to 4.

For specimens which were half immersed, the depth of deterioration of the immersed portion appears to be about as great as for those completely immersed, although for some of the half-immersed specimens disintegration has proceeded above the immersion line. Loss in weight of the half-immersed specimens, also, is more than half that of the completely immersed specimens. There appears to be no distinct advantage in half immersion.

The "daily cycle," consisting of immersion of the specimens in sodium-magnesium sulfate solution for 16 hr followed by drying in circulating air at 130 F for 8 hr, produced results which were in disagreement with those obtained from the other two types of exposure. The effect of this cycle was quite extreme since it involved the rigors of wetting and drying as well as those of sulfate attack. Test results obtained were completely inconsistent and no conclusions could be drawn.

Sodium Sulfate.—Specimens immersed in 2.1 per cent sodium sulfate solution were put through four-week cycles (27 days of immersion followed by 1 day of drying at 70 F and 50 per cent relative humidity). This exposure did not appear to be severe enough to produce distinctive disintegration of the specimens up to a period of 18 months. Considering the 28-day curing period, the only specimens seriously affected were those containing the plain high C_3A cement, and therefore no definite comparison could be made among the various pozzolans.

Type of Disintegration.—The type of sulfate solution to which a concrete was exposed had a definite effect on the type of disintegration. The sodium-magnesium sulfate solution tended to attack all of the exposed surfaces of the concrete and to cause fairly uniform scaling, flaking, or light spalling. The sodium sulfate solution, on the other hand, tended to penetrate deeply into the concrete, particularly at each end of a specimen, causing swelling, cracking, and spalling of large pieces of concrete from the ends without spalling or any other apparent damage to the side surfaces of a specimen. Also, it seemed that the sodium sulfate portion of the mixed sulfate solution tended to attack and penetrate into specimens containing the high C_3A cement causing rapid disintegration from internal action before any serious surface scaling or spalling could begin. However, for the very low C_3A cement, having high resistance to sulfate action, the disintegration in the mixed solution was by scaling of all surfaces of the specimen.

DISCUSSION OF METHODS OF EVALUATING SULFATE ATTACK

Visual Observation:

Visual observation of specimens was relied upon to a considerable extent to indicate whether it was necessary to take measurements of loss in weight or change in length as frequently as the regularly scheduled intervals of 28 days. The photographic record supporting visual observation was useful in studying and analyzing the data obtained from the other methods of evaluating sulfate attack. The main function of the photographic record, however, was to provide a visual history of the progress of disintegration for each specimen selected.

As a practical matter it is recognized that although a photographic record would be very satisfactory, it would be an expensive procedure and probably not feasible in many investigations.

Loss in Weight:

The loss-in-weight method seemed to be the most reliable, in this investigation, for evaluating the resistance of concretes to sulfate attack. Because of the difference in behavior of specimens in each of the two types of solution, the weighing of specimens at scheduled intervals and at intermediate times (as found necessary) gave a more definite indication of disintegration than did change in length. In some cases the values from the loss-in-weight method were more indicative of actual degree of disintegration than values from the change in compressive strength method. The actual determination of the loss in weight of each representative specimen selected for photographing gave a close check on the disintegration as shown by visual observation.

Although long periods of sulfate exposure are necessary, it is felt that this method of evaluating disintegration is the simplest of the four methods employed in this investigation.

Change in Length:

The change-in-length method of evaluating resistance to sulfate attack was the least satisfactory of the four methods employed. Specimens immersed in the sodium sulfate solution, and which disintegrated quite rapidly because of penetration of the sulfate into the specimen, expanded to considerable degree. This expansion produced a definite movement of the gage plug embedded at the center of each end of the specimen. However, for specimens immersed in the sodium-magnesium sulfate solution, and which disintegrated primarily by surface scaling and light spalling, the measurement of length change probably was far from a true indication of the disintegration because this sulfate solution did not penetrate deeply enough into the interior to cause axial expansion of the specimen. Therefore, the gage plug embedded in each end of the specimen changed its position very little or else very slowly, and a positive indication of disintegration was not obtained. For these reasons, it is felt that this method of evaluation would not be considered satisfactory where disintegration occurs by surface scaling rather than by swelling.

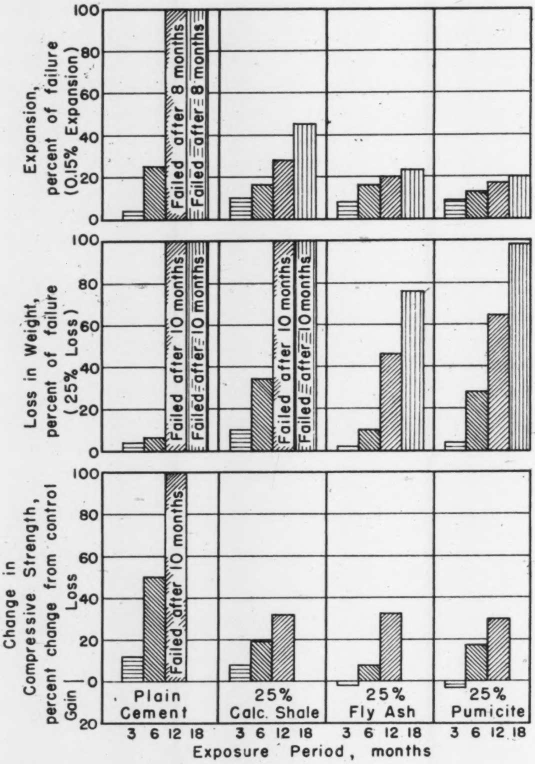

FIG. 7.—Sulfate Attack as Evaluated by Expansion, Weight Loss and Compressive Strength of High C_3A Cement-Concretes with Pozzolan.

Complete immersion (after curing for 28 days) in 5 per cent sodium plus 5 per cent magnesium sulfate.

Compressive Strength:

The compressive strength method of evaluating resistance to sulfate attack gave an indication of the degree of disintegration that was not as definite as that obtained by the loss-in-weight method. Although results obtained by these two methods agreed reasonably well where disintegration was extensive, the compressive strength method failed to distinguish small differences. Since the strength obtained is that for the remaining fairly sound portion of a treated specimen, it does not truly indicate an absolute value of the degree of disintegration. This relation would be especially true for concretes immersed in the sodium-magnesium sulfate solution, which disintegrated primarily by surface scaling. However, the compressive strength method did give more consistent results than the change-in-length method.

SUMMARY

A comparison of results obtained with the three methods of evaluating degree of disintegration is afforded by Fig. 7. Concretes of this figure contained the high C_3A cement, either plain or with pozzolan, and were cured 28 days prior to immersion in sodium-magnesium sulfate solution. For the plain portland cement, all three methods (loss in weight, change in length, and change in compressive strength) indicated approximately the same degree of disintegration. For specimens containing the pozzolans, it is felt that the only true indicator of potential sulfate resistance is loss in weight, because results by this method are in close agreement with the visual observation of degree of deterioration.

The diagrams for change in length show very small expansion for specimens which by the loss-in-weight method are approaching failure. Likewise, the diagrams for compressive strength indicate only partial disintegration of specimens which by the loss-in-weight method are approaching failure.

To summarize, it seems that neither the change-in-length nor the change in compressive strength method was as satisfactory an indicator of resistance to sulfate attack as was the loss-in-weight method.

CONCLUSIONS

From the results of the tests reported herein, the following conclusions appear to be justified.

1. A pozzolan of high quality used as a partial replacement of high C_3A cement will produce concrete having greater sulfate resistance than plain high C_3A cement concrete.

2. The use of a very low C_3A cement results in concretes having superior resistance to sulfate action, with practically no disintegration under any of the conditions of these tests. A partial replacement of the very low C_3A cement with pozzolan produces no improvement in sulfate resistance.

3. Extending the period of curing beyond 28 days has little or no effect in improving or increasing resistance of concretes to the action of a mixture of sodium and magnesium sulfate solutions. A long curing period appears to have an adverse effect on concretes containing high C_3A cement, either alone or with calcined shale, which are exposed to sodium sulfate attack.

4. Continuous complete immersion of concrete specimens in sulfate solutions appears to be a very satisfactory exposure for laboratory investigations. Although results obtained on concrete specimens only half immersed in sulfate solution agree with those obtained on specimens fully immersed, the more complex laboratory procedure involving half immersion makes this exposure method impractical. The daily cycle of immersion in sulfate solution, followed by drying in an oven at 130 F did not accelerate disintegration as had been expected. Results obtained with this exposure were questionable and attempted analysis indicated them to be too inconsistent to report herein.

5. A mixture of sodium-magnesium sulfate attacks the exposed concrete surfaces, causing scaling or spalling of the mortar, whereas the sodium sulfate alone tends to penetrate into the concrete specimen, causing swelling, cracking, and spalling of large pieces of concrete without scaling of the exposed surface. Pozzolans exhibited their best performance in concretes exposed to sodium sulfate attack rather than to the mixture of sodium-magnesium sulfate.

6. Of the several methods employed for measuring the rate of deterioration of concrete, the loss-in-weight of specimens seems to provide the most positive evaluation. The change-in-length method as used in this investigation results in data which generally are in accord with those obtained by loss-in-weight determinations but nevertheless contain many inconsistencies. Similar inconsistencies are observed with the change-in-compressive-strength method. Visual observation and examination of photographs of representative specimens provides a very satisfactory criterion for qualitatively evaluating degree of sulfate attack but might not be practical for many investigations because of the high cost.

Recommendations:

The results of this investigation point up the need for an accelerated reliable method of evaluating potential sulfate resistance of concretes containing portland cement and pozzolan. The Working Committee on Sulfate Resistance of ASTM Committee C-1 has developed a performance test for the potential sulfate resistance of portland cement (**4**). This method consists in measuring the expansion of mortar prisms containing excess calcium sulfate, equivalent to a total SO_3 content of 7.0 per cent by weight of the cement. The expansion of these prisms stored in water is an index of the potential sulfate resistance of the cement. This same test procedure should be evaluated with mortar bars containing pozzolan as partial replacement of portland cement.

The physical tests of this investigation clearly indicate that concretes containing the high C_3A cement and pozzolan exhibit markedly higher sulfate resistance than do concretes containing the high C_3A cement without pozzolan. A physicochemical study of the mechanism by which pozzolans improve the sulfate resistance should be made in order to provide basic information on the pozzolanic reaction and its effect on sulfate resistance.

Acknowledgments:

In carrying out this investigation, the advice and help of a number of individuals were sought. The authors wish to express their appreciation to all who had a part in this work.

Those who contributed much to this program and who deserve special mention are Cecil H. Best, Guillermo Castellanos, Norman E. Haavik, and George Hayler. The authors wish to thank R. E. Davis for his helpful advice and J. W. Kelly for his critical review of the manuscript.

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DISCUSSION

MR. C. M. WAKEMAN.[1]—Would the same data apply to specimens stored in sea water or have such tests in sea water been undertaken?

MR. MILOS POLIVKA (*author*).—I am quite sure that these data would also apply to concretes exposed to sea water except that the concentration of sulfates is very weak and what we are trying to do here is to accelerate sulfate attack as much as possible.

MR. M. A. SWAYZE.[2]—It is most gratifying to find someone showing that pozzolans do not always work wonders. The unqualified claim has been made so many times in recent years that "pozzolans increase the sulfate resistance of portland cements" that I have been coming slowly to the boiling point. Consequently I am most happy to have this author's paper, and to comment on it.

Good pozzolans do improve the poor sulfate-resistant properties of cements high in C_3A content, largely by dilution of this reactive component, but not to the point where they will insure long life of concrete in a sulfate exposure. On the other hand, the same pozzolans can literally wreak havoc with cements in which the C_3A content is either very low or absent, and which are highly resistant in their own right to sulfate action. Our research laboratory has data on a considerable number of pozzolans, and this differing action was found for each of them, when tested with cements with high and with low C_3A contents.

The reason for this difference in action may be seen if we carry our theoretical chemistry a little further than is commonly done. The most common claim for pozzolans is that "they combine with the hydrated lime (sometimes erroneously called free lime) which is liberated during the hydration of portland cement." This statement like the one on improvement in sulfate resistance, is only a half-truth, since even the best pozzolans rarely combine with as much as 50 per cent of the hydrated lime normally present in a saturated cement paste within a year's time. The general assumption is that the hydrated lime reacts only with the silica portion of the pozzolan. The question of what happens to the usually large amount of alumina present in pozzolans while this lime-silica combination is going on has apparently never been asked.

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The fact is that alumina, once it is freed from its combination with silica, will react with hydrated lime to form C_3A hydrate even more readily than in the similar lime-silica combination.

Either of these reactions with hydrated lime is very slow in comparison with the relatively rapid reaction of portland-cement grains with water. This is easy to understand, since pozzolan grains, unhydrated cores of cement grains, and the minute crystals of hydrated lime resulting from cement hydration are all encased in the rather impermeable gel set up by the hydrating cement. The only chance for pozzolanic action is by diffusion through this gel structure.

In consequence, the increase in quantity of the C_3A hydrate due to pozzolanic action is slow. With blends of pozzolan with high C_3A portland cements, this late effect is masked by early prolongation of resistance due purely to dilution of the cement's C_3A content. With low C_3A cement blends, no such masking occurs, since the straight cement is already highly resistant to sulfate action. In this case, assuming the pozzolan to be a good one and that it has the usual high alumina content, the behavior of the portland-pozzolan blend in sulfate exposure will become progressively worse as time goes on. Differences will be noted within a year, as the author has found. Greater differences will be found at later ages. I therefore hope that the author still has his original comparative specimens and will carry on his observations with them for at least a few more years.

In this writer's estimation, no cement or blend deserves to be called moderately or highly sulfate resistant unless the life of mortars and concretes made with it and subjected to moderate or severe sulfate exposures is practically as long as in fresh water. The author has already brought us a long way toward clearer thinking on the role of pozzolans in concrete. For this he deserves our thanks. However, tests for comparative sulfate resistance cannot be hurried. I therefore hope that he will continue on this important subject, to the end that the current superstition to the effect that "pozzolans increase the sulfate resistance of portland cements" can be discarded.

LONG-TIME CREEP AND SHRINKAGE TESTS OF PLAIN AND REINFORCED CONCRETE*

BY G. E. TROXELL,[1] J. M. RAPHAEL,[2] AND R. E. DAVIS[3]

SYNOPSIS

A comprehensive investigation of creep and drying shrinkage of concrete has extended over a period of nearly 30 yr. Many of the specimens were 4-in. diameter unsealed cylinders loaded in compression to 800 psi at 28 days and thereafter stored in air at 50 per cent relative humidity. Both creep and shrinkage continued during the entire period. Eighteen to 35 per cent of the total creep during 20 yr occurred in the first 2 weeks of loading and 64 to 83 per cent within 1 yr. Fourteen to 34 per cent of the total shrinkage during 20 yr occurred in the first 2 weeks of drying and 66 to 85 per cent within 1 yr. Creep increased with a higher water-cement ratio and with a lower aggregate-cement ratio, but shrinkage was not appreciably affected by either of these variables. Both creep and shrinkage of concrete were greater for type IV than for type I cement. The creep for sandstone concrete was more than double that for limestone concrete, and the shrinkage of sandstone concrete was more than double that for quartz concrete.

For ages of loading from 28 to 90 days, the older the specimen at time of loading, the less the creep. For stresses from 300 to 1200 psi, the average creep per unit of stress was slightly greater at the high stresses than at the low stresses but for each increment of higher stress the corresponding increment of creep per unit stress was significantly greater. The moisture conditions of storage had a great effect on both creep and shrinkage; the creep for storage in air at 50 per cent relative humidity was about 3 times that for storage in water and the shrinkage at 50 per cent relative humidity was about 1.4 times that at 70 per cent relative humidity and about 8 times the swelling for storage in water. For specimens having a diameter of 4 to 10 in. under moist storage, the creep decreased as the size of specimen increased. For the range of conditions covered by the investigation, the combined shrinkage or swelling and creep varied from about 1 to 11 times the instantaneous deformations.

Small concrete columns of 2000-psi concrete reinforced with 1.9 and 5 per cent of steel, and of 4000-psi concrete reinforced with 1.9 per cent of steel, were stored in air, with corresponding columns in water, to determine the effect of these conditions on the stresses developed in the concrete and steel while under load. The test results show that stress was transferred from the concrete to the steel, until, in a few cases, the concrete was actually in tension and the steel was stressed to the yield point in compression.

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* Presented at the Sixty-first Annual Meeting of the Society, June 22-27, 1958.

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[3] Professor of Civil Engineering and Director of Engineering Materials Laboratory (Emeritus), University of California, Berkeley, Calif.

The results of tests by many investigators have shown that concrete is a material which, when subjected to sustained stress, continues to yield or to deform over a considerable period of time. The property of yielding with time, previously designated as "plastic flow," is now commonly called creep. It includes only those deformations which occur as a result of application of load. Concrete also shrinks upon drying and swells upon wetting.[4]

This paper describes several test series made at the University of California, some of which were started as early as 1926, so that some specimens have been under observation for nearly 30 yr. These tests were designed to show the compressive creep and the shrinkage with time as affected by:

(*a*) Aggregate-cement ratio;
(*b*) Water-cement ratio;
(*c*) Composition and fineness of cement;
(*d*) Mineralogical character of aggregate;
(*e*) Maximum size of aggregate;
(*f*) Magnitude of compressive stress;
(*g*) Moisture conditions of storage;
(*h*) Size of specimen;
(*i*) Age and duration of loading; and
(*j*) Steel reinforcement.

Earlier results of these tests have been analyzed and conclusions published in several papers (**1–4, 6**).[5] In this paper, a fresh look has been taken at the results of the tests, which now have run nearly thirty years. No major contradictions to the earlier conclusions have appeared. What is unique is that creep and shrinkage, under the conditions of the reported tests, have been observed to continue over the entire period of observation, although at a greatly decreased rate at the later ages.

It is believed that these test results can be used as a new yardstick by which to measure "ultimate creep" from "short-time" tests of a year or so.

SCOPE OF TESTS

Plain Concrete:

The tests on plain concrete reported herein are divided into five series as follows:

Series 2 was inaugurated in 1926 to determine the effect of magnitude of unit stress and age at time of loading on creep and shrinkage. Variables for this series were age at time of loading and intensity of compressive loading. All specimens employed the same aggregate and cement, with constant aggregate-cement ratio, water-cement ratio, slump, curing to the age of loading and storage thereafter.

Series 3 was inaugurated in 1928 to determine the effect of moisture conditions of storage on creep and shrinkage. All specimens for this series employed the same aggregate and cement and were of the same aggregate-cement ratio, water-cement ratio, curing to age of loading, and intensity of loading. After loading, storage for all specimens was at 70 F, but the moisture conditions of storage were varied.

Series 4 was inaugurated in 1928 to determine the effect of the mineralogical character of the aggregates on creep and shrinkage. For this series, one cement and six different types of aggregate were used in concretes having a constant aggregate-cement ratio and water-cement ratio. All specimens were subjected to the same curing conditions and intensity of loading.

Series 13 was inaugurated in 1932 to determine the effect of size of aggregate and size of specimen on creep and shrink-

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[4] Shrinkage and swelling relate to moisture change whereas contraction and expansion are the results of thermal change.

[5] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 1120.

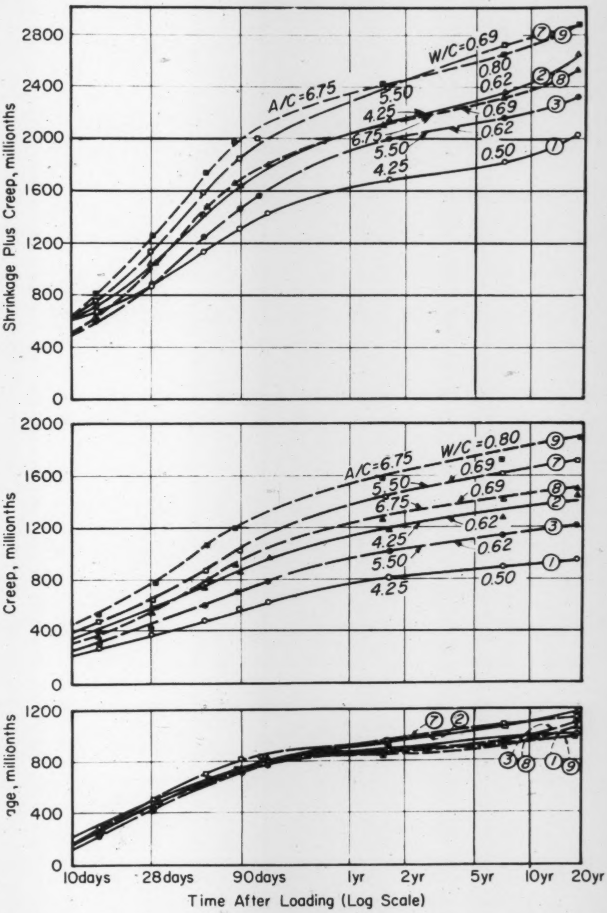

FIG. 1.—Effect of Aggregate-Cement Ratio and Water-Cement Ratio upon Creep and Shrinkage.

Series 17.—Specimens, 4- by 8-in. cylinders. Aggregate, 0- to ¾-in. local gravel. Fineness modulus, 4.94. Cement, type I; Wagner specific surface, 1300 sq cm per g. Consistency, variable. Water content, pcy: group 1—342; group 2—404; group 3—341; group 7—372; group 8—322; group 9—363. Curing in fog at 70 F for 28 days. Age at loading, 28 days. Sustained compressive stress, 800 psi. Storage after loading, in air at 70 F and 50 per cent relative humidity. Instantaneous deformations in millionths: group 1—243; group 2—305; group 3—255; group 7—401; group 8—235; group 9—341.

age. In all tests of this series, the aggregate-cement ratio, water-cement ratio, curing, and intensity of loading were held constant. For the first part of the series, the size of the specimen was held constant and the fineness modulus of the aggregates was varied. For the second part of the series, the fineness modulus of the aggregates was held constant and the cylinder size was varied.

Series 17 was inaugurated in 1934 to determine the effect of richness of mix, water-cement ratio, and composition and fineness of cement on creep and shrink-

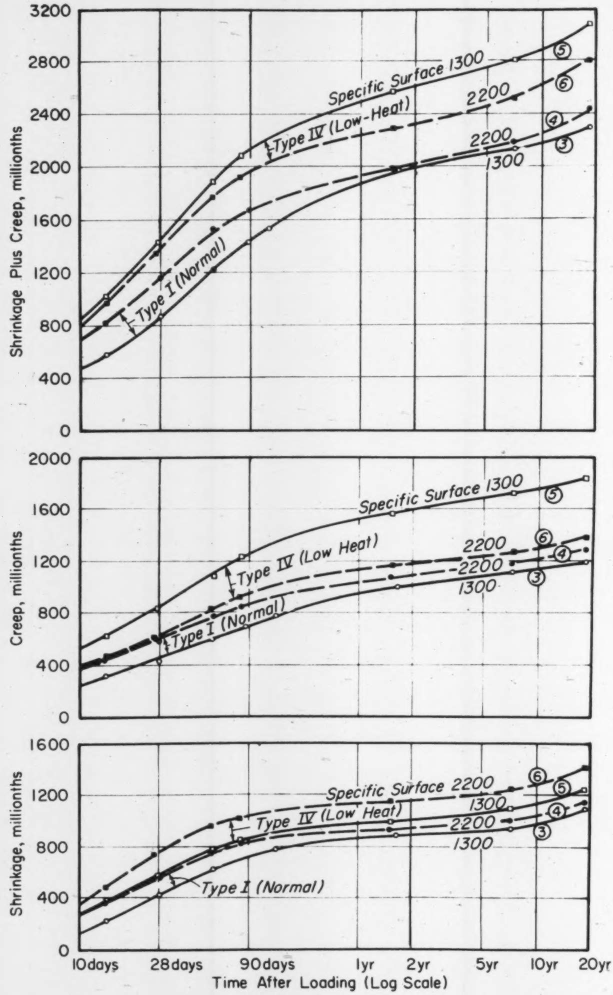

FIG. 2.—Effect of Composition and Fineness of Cement upon Creep and Shrinkage.

Series 17.—Specimens, 4- by 8-in. cylinders. Aggregate, 0- to ¾-in. local gravel. Fineness modulus, 4.94. Cement, (*a*) type I, 52 per cent C_3S, 25 per cent C_2S, 8 per cent C_3A, 13 per cent C_4AF, and (*b*) type IV, 30 per cent C_3S, 47 per cent C_2S, 8 per cent C_3A, 12 per cent C_4Af. Specific surface, Wagner. Aggregate-cement ratio by weight, 5.50. Water-cement ratio by weight, 0.62. Consistency, variable. Curing, in fog at 70 F for 28 days. Age at loading, 28 days. Sustained compressive stress, 800 psi. Storage after loading, in air at 70 F and 50 per cent relative humidity. Instantaneous deformations in millionths: group 3—255; group 4—288; group 5—313; group 6—246.

age. In this series, the variables were the consistency, aggregate-cement ratio, water-cement ratio, and type and fineness of the cement, but curing and storage conditions and the intensity of loading were constant.

Reinforced-Concrete Columns:

A series of tests was begun in 1931 to determine the progressive changes in

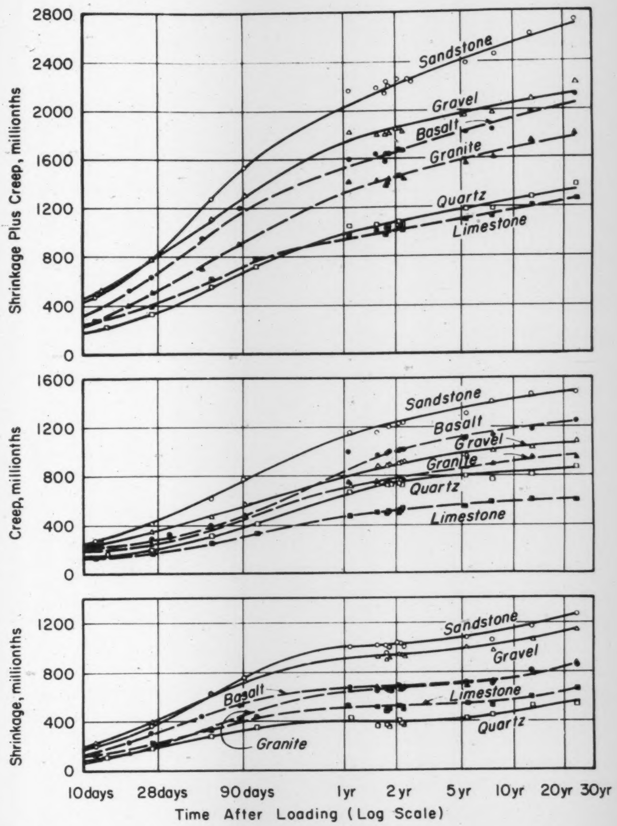

FIG. 3.—Effect of Mineralogical Character of Aggregates upon Creep and Shrinkage.

Series 4.—Specimens, 4- by 14-in. cylinders. Fineness modulus 5.75, except gravel which had fineness modulus 5.61. Cement, type I. Aggregate-cement ratio by weight, 5.67. Water-cement ratio by weight, 0.59. Curing, 28 days in fog at 70 F. Age at loading, 28 days. Sustained compressive stress, 800 psi. Storage after loading, in air at 70 F and 50 per cent relative humidity. Instantaneous deformations in millionths: sandstone, 281; gravel, 275; basalt, 224; limestone, 222; quartz, 212; granite, 205.

length and stresses due to shrinkage and creep in reinforced concrete columns. Specimens were plain and reinforced concrete columns, using two strengths of concrete, two percentages of longitudinal steel, and two percentages of spiral steel. All specimens were cured in fog at 70 F for 3 weeks; thereafter some were stored in air and some in water. Loads were applied at 28 days.

TESTING APPARATUS AND METHODS

The majority of the specimens of each series were subjected to sustained compressive stresses. These stresses were maintained constant by use of one or more coil springs held in compression by a system of rods and plates between which the specimen was clamped (1).

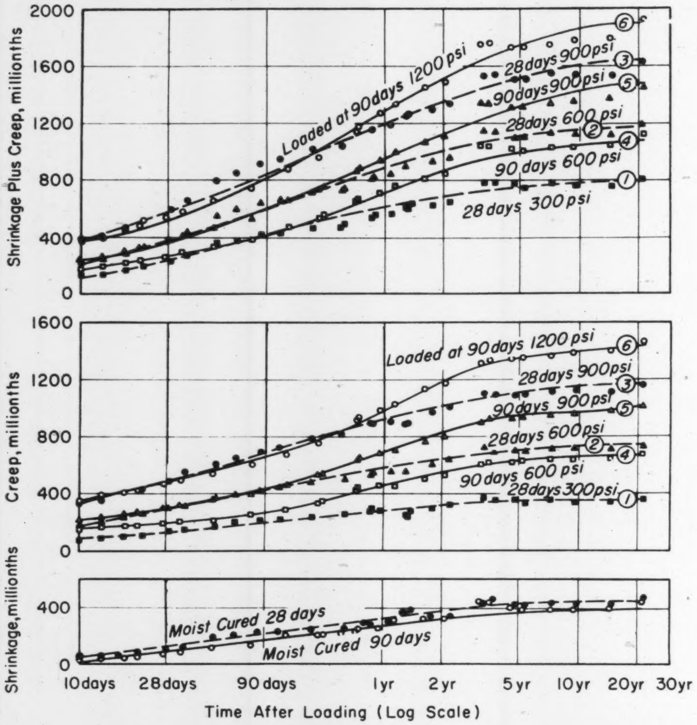

FIG. 4.—Effect of Age and Intensity of Loading upon Creep and Shrinkage.

Series 2.—Specimens, 4- by 14-in. cylinders. Aggregate, 0- to 1½-in. crushed granite. Fineness modulus, 5.03. Cement, type I. Aggregate-cement ratio by weight, 5.05. Water-cement ratio by weight, 0.69. Slump, 3 in. Curing, moist at 70 F until time of loading. Sustained stress, compression, as indicated. Storage after loading, in air at 70 F and 70 per cent relative humidity. Instantaneous deformations in millionths: group 1—90; group 2—285; group 3—620; group 4—190; group 5—340; group 6—515.

The desired load was applied by a small portable hydraulic testing machine installed in a room where both air temperature and humidity were controlled. After loading in the machine, the nuts on the tension rods of the load-sustaining apparatus were brought up against the end plates. Upon releasing the load on the machine, the compression in the coil spring and in the specimen was maintained by tension in the rods.

Auxiliary or control specimens subjected to the same curing and storage conditions as the loaded specimens, but under no stress, have been under observation in order to determine the changes in length (shrinkage or swelling) due to causes other than stress. The magnitude of creep was taken as the difference between the changes in length for the loaded specimens and the corresponding unloaded control specimens.

Observations of changes in length of the loaded as well as the unloaded control specimens were made by use of a 10-in. fulcrum-plate strain gage, the strain-gage inserts in the concrete being of stainless steel screwed into brass plugs cast in the specimens. Each cylindrical specimen had three longitudinal gage lines 120 deg apart.

TEST RESULTS

The test results for the plain concrete are shown in Figs. 1 to 10 inclusive. Excepting Figs. 5, 9, and 10, the lower part of each figure shows the shrinkage or swelling as determined by observations on the control specimens, the upper part shows the total length change due to shrinkage or swelling and creep as determined by observations on the loaded specimens, and the middle part shows the creep for the loaded specimens as determined by the difference between the corresponding curves in the upper and lower parts of each figure. In general, all figures show that both shrinkage and creep increase with time, although at a very low rate at the later ages. For the average of all test conditions, about two thirds of the total shrinkage and one half of the total creep occurred within the first 3 months of testing.

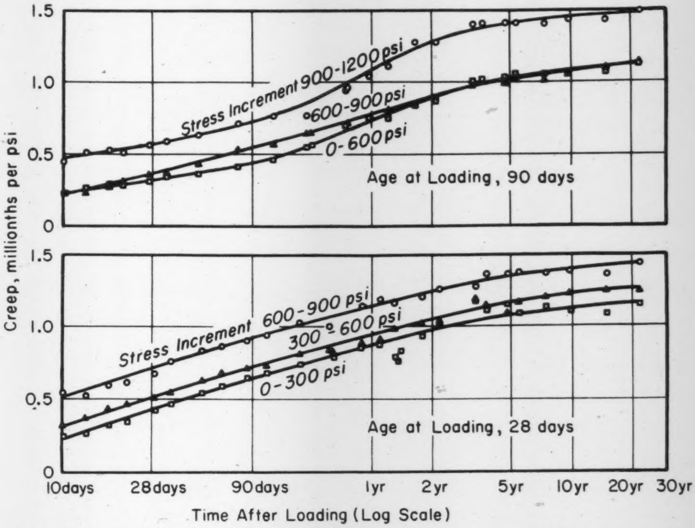

FIG. 5.—Effect of Level of Stress Increment upon Creep—Creep Data Taken from Fig. 4.

In general, after 20 yr the creep ranged roughly from two to six times the so-called instantaneous deformations (observed soon after loading) shown in the notes for each figure; the creep plus shrinkage ranged roughly from three to eleven times the instantaneous deformations.

Effect of Aggregate-Cement Ratio and Water-Cement Ratio:

Figure 1 covers a range of mixes having aggregate-cement ratios from 4.25 to 6.75 by weight and water-cement ratios from 0.50 to 0.80 by weight, giving variable consistencies. For these mixes the shrinkages at a given age were fairly uniform for all conditions. While the water-cement ratio varied from 0.50 to 0.80, the actual water content of all these mixes varied only from 322 to 404 pcy. After 19 yr the shrinkage roughly varied from 1000 to 1200 millionths per unit, but was related neither to the richness of mix, the water-cement ratio, nor the total water content of the mix. The creep

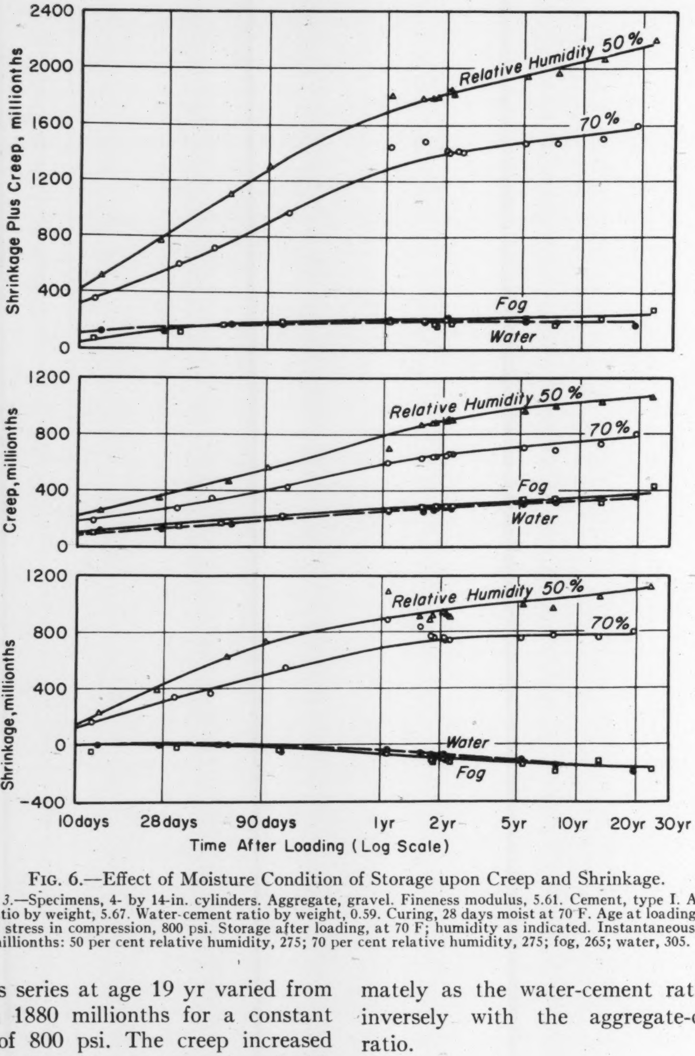

FIG. 6.—Effect of Moisture Condition of Storage upon Creep and Shrinkage.

Series 3.—Specimens, 4- by 14-in. cylinders. Aggregate, gravel. Fineness modulus, 5.61. Cement, type I. Aggregate-cement ratio by weight, 5.67. Water-cement ratio by weight, 0.59. Curing, 28 days moist at 70 F. Age at loading, 28 days. Sustained stress in compression, 800 psi. Storage after loading, at 70 F; humidity as indicated. Instantaneous deformations in millionths: 50 per cent relative humidity, 275; 70 per cent relative humidity, 275; fog, 265; water, 305.

for this series at age 19 yr varied from 950 to 1880 millionths for a constant stress of 800 psi. The creep increased with a higher water-cement ratio and with a lower aggregate-cement ratio but did not correlate directly with total water content of the mix.

The combined shrinkage plus creep for this series at age 19 yr varied from 2000 to 2850 millionths, and it varied approximately as the water-cement ratio and inversely with the aggregate-cement ratio.

Effect of Composition and Fineness of Cement:

Series 17, used to evaluate the effect of composition and fineness of cement, included both type I (normal) and type

IV (low-heat) portland cements with Wagner specific surfaces of 1300 and 2200 sq cm per g. The aggregate-cement ratio (using a ¾-in. maximum size of gravel) was 5.50 by weight and the water-cement ratio was 0.62 by weight. The slump of the concrete was variable.

From the test results for series 17 shown in Fig. 2, it is evident that the shrinkage was still continuing at a slow rate even at age 19 yr, when the shrinkages varied from 1100 to 1420 millionths, although about two-thirds of this occurred in the first 3 months. The shrinkage for the low-heat (type IV) cement was consistently greater than for the normal (type I) cement, and greater for the fine cement than for the coarse. For the cements having a Wagner specific surface of 2200, the shrinkage of the low-heat cement concrete was from 200 to 270 millionths greater than for the normal cement concrete, whereas for a specific surface of 1300 the difference was consistently about 150 millionths. For the finer grind of normal cement, the concrete shrinkage at 1 month was about 120 millionths greater than for the coarse grind; but at age 19 yr the difference was only 50 millionths. For the low-heat cement the difference due to fineness of grinding was consistently about 200 millionths.

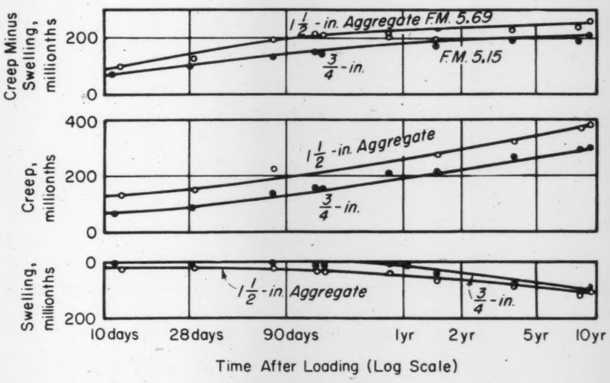

FIG. 7.—Effect of Size of Coarse Aggregate upon Creep and Expansion While in Moist Storage.

Series 13.—Specimens, 6- by 12-in. cylinders. Aggregate, gravel. The ¾-in. maximum mix was obtained by substituting for the ¾- to 1½-in.-aggregate used in the 1½-in. maximum mix, an equal solid volume of ¼- to ¾-in. aggregate. Cement, type I. Aggregate-cement ratio by weight, 6.95. Water-cement ratio by weight, 0.61. Curing, 28 days moist at 70 F. Age at loading, 28 days. Sustained stress in compression, 800 psi. Storage after loading in fog at 70 F. Instantaneous deformations in millionths: ⅜- to 1½-in., 238; ⅜- to ¾-in., 212.

The creep in this series varied from 1200 millionths for the coarse normal cement to 1850 millionths for the coarse low-heat cement, but it was still continuing slowly even at age 19 yr. In general, about one-half to two-thirds of this creep occurred within the first 3 months. The creep for the low-heat cement was consistently greater than for the normal cement. For the low-heat cement, the creep for the coarse grind was about 30 per cent greater than for the fine grind, but for the normal cement the creep for the fine grind was somewhat greater than for the coarse grind. It is believed that this may be due to variations in the particle size distribution of the two cements, which are not apparent in the calculated values of specific surface.

Where drying shrinkage is not an item of importance, as in mass structures, concretes containing coarse-ground low-heat cement, which is more susceptible to creep, will have the lesser tendency towards cracking. The results of extensibility tests have demonstrated the truth of this statement. (Coarse-ground, low-heat cements were used in the construction of Crystal Springs Dam and Morris Dam, both of which have enjoyed general freedom from cracking.)

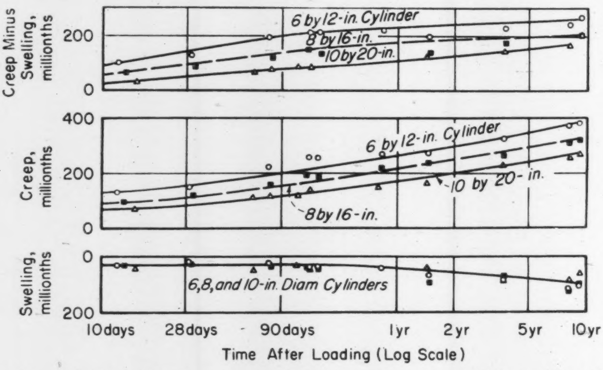

FIG. 8.—Effect of Size of Specimen upon Creep and Expansion While in Moist Storage.

Series 13.—Specimen height, 2 diameters. Aggregate, gravel. Fineness modulus, 5.69. Cement, type I. Aggregate-cement ratio by weight, 6.95. Water-cement ratio by weight, 0.61. Curing, 28 days moist at 70 F. Age at loading, 28 days. Sustained stress in compression, 800 psi. Storage after loading, fog at 70 F. Instantaneous deformations in millionths: 6-in. cyl., 238; 8-in. cyl., 239; 10-in. cyl., 248.

The combined shrinkage and creep for this series varied from 2300 to 3100 millionths, being greatest for the coarse low-heat cement and least for the coarse normal cement. It ranged from nine to eleven times the corresponding instantaneous deformation.

Effect of Mineralogical Character of Aggregate:

Series 4 covered the effect of the mineralogical character of the aggregates on creep and shrinkage. Five groups of specimens were made, in which the fine and coarse aggregate of each group was composed entirely of one rock type; these included quartz, limestone, granite, basalt, and sandstone. For a sixth group the aggregate was a local sand and gravel containing a variety of siliceous minerals, principally graywacke and other sandstones. The aggregate grading was the same for all groups.

The shrinkage in this series at age 23 yr, as shown in Fig. 3, ranged in millionths as follows: quartz, 550; limestone, 650; granite and basalt, 850; gravel, 1140; and sandstone, 1260. All shrinkages were increasing slowly even at this late age although about one-half to two-thirds of the shrinkages at 23 yr occurred within the first 3 months.

The corresponding creep values ranged in millionths as follows: limestone, 600; quartz, 860; granite, 960; gravel, 1070; basalt, 1250; and sandstone, 1500. Depending upon the mineralogical character of the aggregate, the maximum creep was $2\frac{1}{2}$ times the minimum. At 23 yr all creep values were still increasing slowly although about one-half of the creep at 23 yr occurred within the first 3 months.

The combined shrinkage and creep ranged in millionths as follows: limestone, 1260; quartz, 1340; granite, 1780; basalt, 2060; gravel, 2180; and sandstone, 2720. About one half of the shrinkage and creep occurred within the first 3 months. As the materials in each mineralogical group may vary, such variations can be expected to affect their shrinkage and creep characteristics.

The instantaneous deformations were greatest for the concretes containing sandstone and gravel aggregates, least for those containing granite and quartz, and intermediate for basalt and limestone. While there are some discrepancies, the long-time deformations, both in creep and shrinkage, appear to be roughly proportional to the instantaneous deformations, so that creep and shrinkage vary inversely with the modulus of elasticity at the time of loading. The combined shrinkage and creep ranged roughly from six to nine times the instantaneous deformations, this factor being lowest for limestone and quartz, and highest for sandstone and basalt.

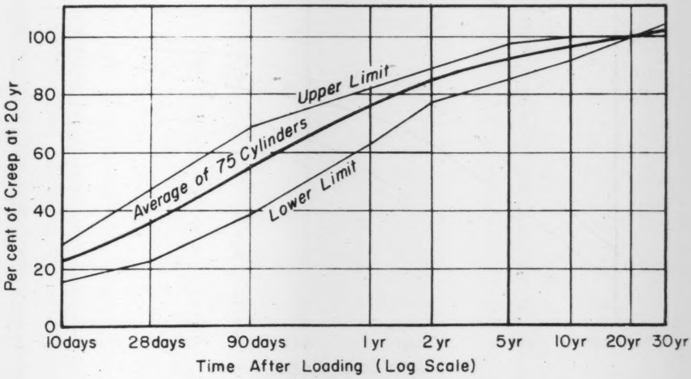

Fig. 9.—Creep-Time Ratio Curves.

20-yr values taken as 100. Creep data taken from Figs. 1, 2, 3, 4, 6. Cylinder sizes, 4 by 8 in. and 4 by 14 in. Maximum aggregate, ¾ and 1½ in. Aggregate types, several. Cement, types I and IV, both fine and coarse. Aggregate-cement ratio by weight, 4.25 to 6.75. Water-cement ratio by weight, 0.50 to 0.80. Age at loading, 28 and 90 days. Sustained compressive stress, 300 to 1200 psi. Relative humidity while loaded, 50 to 100 per cent.

Effect of Age of Concrete at the Time of Loading:

Figure 4, giving the results of tests on series 2, shows the effect of age of concrete at time of loading and the effect of intensity of load on creep and shrinkage.

Only two ages at loading were chosen—28 days and 90 days. It can be seen that for the same intensity of load, the earlier the concrete was loaded, the greater was the resulting creep. After 1 yr of loading, the creep for the cylinders loaded at age 90 days was about 20 per cent less than for the cylinders loaded at age 28 days, but the difference in the short-time creep was much greater. (In tests of sealed specimens initially loaded at various ages up to 5 yr, Hanson (5) has shown that the resulting creep rate drops off rapidly with age at time of loading up to about 10 days and subsequently the effect of age at time of loading on creep is less well marked.)

The shorter the curing period the greater the shrinkage, though the effect of length of curing period was not large. In these tests, shrinkages were all relatively low because of storage at the relatively high humidity of 70 per cent.

For the concretes loaded at the age of 28 days, the combined shrinkage and creep ranged from about three to four times the instantaneous deformations; for those loaded at 90 days this value ranged from four to six. In each case the high value was for a stress of 600 psi and the low value was for a stress of 900 psi.

Effect of Intensity of Loading:

As shown for series 2 in Fig. 4, the creep after 21 yr was 370 millionths for specimens loaded to 300 psi at 28 days, 740 and 680 millionths when loaded to 600 psi at 28 and 90 days, 1160 and 1000 millionths when loaded to 900 psi at 28 and 90 days and 1450 millionths when loaded to 1200 psi at 90 days. For the three intensities of loading at age 28 days, the creep was almost directly proportional to the load; this is also approximately true for the loadings at age 90 days.

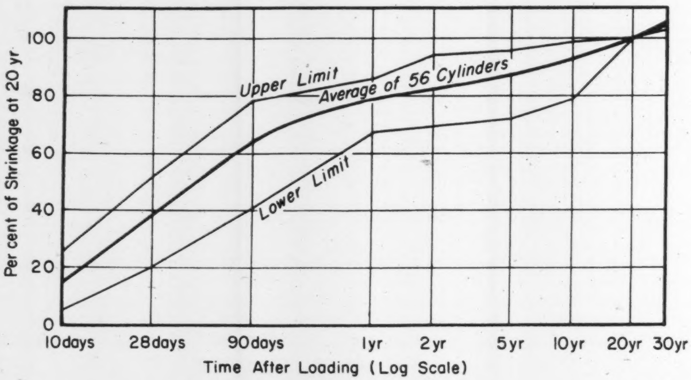

FIG. 10.—Drying Shrinkage-Time Ratio Curves.

20-yr values taken as 100. Shrinkage data taken from Figs. 1, 2, 3, 4, 6. Cylinder sizes, 4 by 8 in. and 4 by 14 in. Maximum aggregate, ¾ in. and 1½ in. Aggregate types, several. Cement, types I and IV, both fine and coarse. Aggregate-cement ratio by weight, 4.25 to 6.75. Water-cement ratio by weight, 0.50 to 0.80. Curing period, 28 and 90 days. Relative humidity during storage, 50 and 70 per cent.

The combined shrinkage and creep in 21 yr in this series varied from 800 millionths for the specimens loaded to 300 psi at age 28 days to 1900 millionths for the specimens loaded to 1200 psi at age 90 days. These combined values ranged from three to nine times the instantaneous deformations for the specimens loaded at age 28 days, and ranged from four to six times the instantaneous deformations for those loaded at age 90 days. For each age the lower the stress the higher this factor.

Creep tests at various stress levels are commonly compared by dividing the observed creep by the load intensity to obtain the average creep per unit load; but this tends to obscure the very effect which should be shown. Therefore, the data for Fig. 4 were reanalyzed by a method intended to emphasize the effect of the level of stress on the resulting creep. From the creep at each stress level of 300, 600, 900, or 1200 psi, the creep for the next lower stress level was subtracted, so that a series of curves was produced as shown in Fig. 5 which indicates the creep coefficient at various increments of stress level. It can be seen, from Fig. 5, that the creep per unit stress at the higher levels of stress intensity was significantly greater, with the difference increasing as the stress level became greater.

These stress levels are only a small fraction of the ultimate strength of the concrete, but Shank (7) has shown how creep increases to incipient failure as the stress level approaches the ultimate strength of the concrete. It may thus be expected that increase in magnitude of creep with increasing stress level is a continuous phenomenon from the lowest stress level up to failure.

Effect of Moisture Condition of Storage:

All test results for this series are shown in Fig. 6. After 23 yr storage, the length changes of the unloaded specimens were as follows: In fog or water the swelling was 160 millionths; at 70 per cent and 50 per cent relative humidities the shrinkages were 790 and 1120 millionths respectively. These results show the marked influence of moisture conditions of storage on length changes.

The creep after 23 yr for the loaded specimens were as follows: In water, 360 millionths; in fog, 380 millionths; at 70 and 50 per cent relative humidities the creep was 800 and 1080 millionths respectively. Thus it is seen that within this range the lower the moisture content of the surrounding medium the greater the creep.

The combined shrinkage and creep in this series was 210 millionths for water storage, 250 millionths for fog storage, 1600 millionths for storage at 70 per cent relative humidity, and 2180 millionths for storage at 50 per cent relative humidity. The combined shrinkage and creep at 50 per cent relative humidity was 10 times that for water storage. The combined shrinkage and creep ranged from about 1 to 8 times the instantaneous deformations; the drier the storage condition the higher this value.

Effect of Size of Aggregate:

For these tests the specimens contained gravel aggregate having a fineness modulus of 5.15 for $\frac{3}{4}$-in. maximum aggregate and 5.69 for $1\frac{1}{2}$-in. maximum aggregate. The aggregate-cement ratio by weight was 6.95 for both conditions.

As shown in Fig. 7 the swelling for fog storage was slightly greater for the $1\frac{1}{2}$-in. aggregate than for the $\frac{3}{4}$-in. aggregate, although both values were small in comparison with the shrinkage which would have developed under drying conditions.

At the age of 10 yr the creep values under moist storage conditions were 390 for the $1\frac{1}{2}$-in. aggregate and 300 for the $\frac{3}{4}$-in. aggregate. The comparable instantaneous deformations when first loaded were 212 millionths for the $\frac{3}{4}$-in. aggregate and 238 millionths for the $1\frac{1}{2}$-in. aggregate.

The reason for the greater early-age creep of the concrete containing the larger aggregate is not apparent. The important thing shown by Fig. 7 is that the rate of long-time creep is not appreciably affected by size of aggregate when the paste content is kept constant.

Effect of Size of Specimen:

For this series, the specimens were of diameters of 6, 8, and 10 in. and a height equal to twice the diameter. Curing and storage were in fog at 70 F. Creep specimens were stressed to 800 psi at the age of 28 days.

As shown in Fig. 8, the swelling for fog storage was essentially the same for all three sizes of cylinders and was relatively small in comparison with the shrinkage which would have developed under drying conditions.

At the age of $9\frac{1}{2}$ yr, the creep values for the 6, 8, and 10-in. diameter cylinders were 400, 340, and 280 millionths respectively. These values definitely show that the creep decreases as the size of specimen increases, but a large part of the difference occurs at a very early age. Instantaneous deformations in millionths were as follows: for 6-in. cylinders, 238; for 8-in. cylinders, 239; for 10-in. cylinders, 248. By referring to Fig. 6, which shows the effect of moisture conditions of storage on creep, it will be seen that the creep values noted above would have been much greater for dry conditions of storage. The results shown indicate that under conditions where free escape of surface moisture is possible, the larger the mass of concrete, the smaller will be the creep for the same loading. Any factors which increase resistance to seepage probably will tend to decrease the creep of concrete.

Effect of Duration of Loading on Creep:

Many investigators have been concerned with the effect of time on creep. The typical creep-time ratio curve in Fig. 9, which shows this effect, was constructed by computing for each group of specimens included in Figs. 1 to 6 the per cent of the 20-yr total creep at each plotted time division. Considering that the several test series embrace a variety of conditions of mix, water-cement ratio, consistency, rock type, storage, load intensity and age at loading, the narrow envelope enclosing all the creep curves is rather surprising, but lends some credence to the term "typical creep-time ratio curve."

If the slope of the creep curve on a semilogarithmic plot is termed the "creep coefficient" K, then the total creep ϵ in time t can be expressed as:

$$\epsilon = K \log_e (t + 1)$$

With some idealization, the average creep-time ratio curve of Fig. 9 could be expressed as two straight lines on the semilogarithmic plot as follows:

$$\epsilon = 0.149 \log_e (t + 1) \text{ for } 0 < t < 730 \text{ days}$$

and

$$\epsilon = 0.86 + 0.0665 \log_e (t - 730)$$

for

$$730 < t < \infty$$

where ϵ represents the fraction of the creep at 20 yr. After 2 yr, the creep coefficient was only 45 per cent of the coefficient previous to that time.

It is interesting to note that, in general, one-fourth of the 20-yr creep took place in the first 2 weeks of loading, about half in the first 2 to 3 months, and about three fourths in the first year. The second year contributed another 10 per cent creep, and it then took 18 more years to produce an additional 15 per cent of creep. On the average, in another 10 yr, creep will increase only 2½ per cent. Averages may be misleading, however, since some concretes attained only two-thirds of their 20-yr creep at the end of 1 yr, while others attained five-sixths of it.

If ultimate creep is predicted on the basis of four thirds of the creep at 1 yr, the maximum error will be in the order of only ±15 per cent. If the time when average creep is 50 per cent of the 20-yr creep, which occurs at about 2½ months, is chosen for predicting creep, errors will be much greater, since the creep at 20 yr may range from 150 to 300 per cent of the creep at 2½ months.

It would appear that if a standard creep test is devised, it should be predicated on a duration of loading of not less than 1 yr.

Effect of Time on Shrinkage:

In an attempt to isolate the time element in the drying shrinkage of concrete, all shrinkage test results on 4-in. diameter cylindrical specimens were plotted on a semilogarithmic scale as percentages of the shrinkage at 20 yr, regardless of the individual conditions of ingredients, mix, or curing. Most specimens were stored at 70 F and 50 per cent relative humidity, but some were stored at 70 per cent relative humidity. Results are shown in Fig. 10. The spread of the shrinkage curves is considerably greater than for the similar study made on creep specimens, but the average of 56 cylinders shows a rapid shrinkage rate for the first 90 days on this semilogarithmic plot, and a markedly lower logarithmic rate for the remaining time. On the average about one half the shrinkage occurred in the first 2 months, nearly 80 per cent in the first year, and the remaining 20 per cent in the ensuing 19 yr. An additional 5 per cent occurred between 20 and 30 yr.

Idealizing the average curve of Fig. 10 as two straight lines, the average ratio of shrinkage to time can be expressed shrinkage for the period after 90 days, is only 35 per cent of the value for the period up to that age.

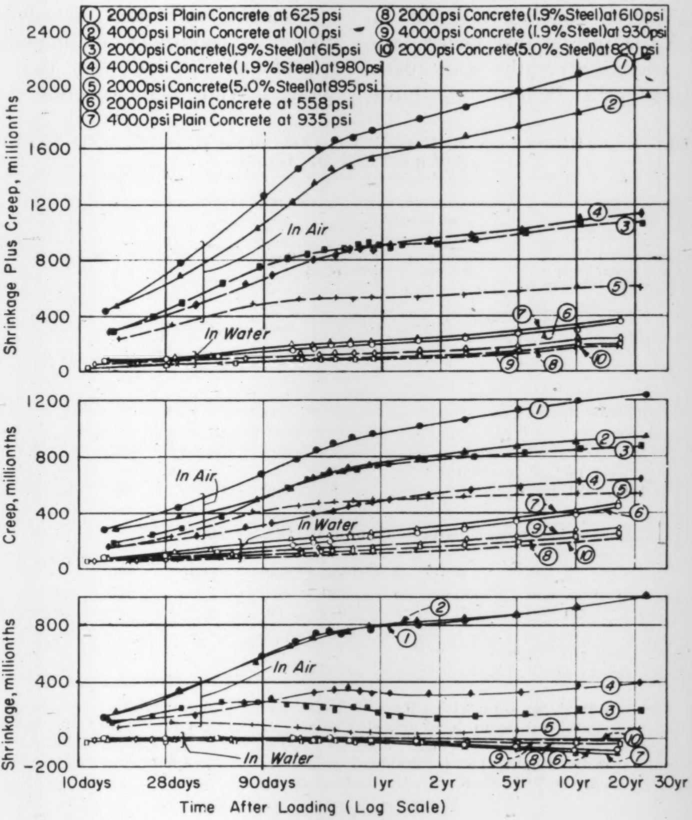

FIG. 11.—Drying Shrinkage and Creep of Plain and Reinforced Concrete Columns. See Table I for column details.

fairly well by the equations

$$\delta = 0.227 \log_e (t + 1) \quad \text{for} \quad 0 < t < 90$$

and

$$\delta = 0.65 + 0.080 \log_e (t - 90) \quad \text{for} \quad 90 < t < \infty$$

where δ is the portion of the total shrinkage at 20 yr. The coefficient of the logarithmic term, representing the rate of

Reinforced Concrete Columns:

Thirty-six round, spirally reinforced concrete columns have been under observation for 22 yr. All columns were reinforced with six evenly spaced longitudinal bars, with either 1.9 or 5.0 per cent of steel. The pitch of the spiral in all cases was 1 in., with the size of the wire so proportioned that the volume of steel in the spiral was one fourth that of the longitudinal steel. The outside diameter of the spiral equalled the diameter of the column so that all the concrete corresponded to "core" concrete as defined in the 1956 Am. Concrete Inst. code.[6] Two nominal concrete strengths were used: 2000 and 4000 psi at 28 days. Two thirds of the total number of columns were stressed, after 28 days initial curing, to the allowable loads computed according to the Joint Committee code of 1924 (8). Half the total number of loaded specimens were stored in air at 70 F and 50 per cent relative humidity and the remainder were stored under water. An additional 16 plain concrete columns and 52 cylinders were used to establish the physical and mechanical properties of the concrete.

TABLE I.—STRESSES IN REINFORCED COLUMNS BEFORE AND AFTER APPLICATION OF LOAD.

Nominal Strength of Concrete at 28 days, psi	Longitudinal Steel, per cent	Total Load Applied to Column, lb	Compressive Stress, psi							
			Before Application of Load		Due to Application of Load		After Application of Load		After Long-Time Loading[b]	
			Steel	Concrete	Steel	Concrete	Steel	Concrete	Steel	Concrete
1	2	3	4	5	6	7	8	9	10	11
COLUMNS STORED IN AIR OF 50 PER CENT RELATIVE HUMIDITY AT 70 F										
2000......	5.0	22 300	1500	−75[a]	9400	895	10 900	820	28 000	−80
2000......	1.9	14 200	900	−20	6400	615	7 300	600	37 800	0
4000......	1.9	21 800	3200	−60	7600	980	10 800	920	42 500[c]	305
COLUMNS STORED UNDER WATER AT 70 F										
2000......	5.0	19 200	0	0	7000	820	7000	820	12 100	555
2000......	1.9	13 650	−300	5	5300	610	5000	615	10 600	505
4000......	1.9	20 600	−700	15	7100	930	6400	945	13 400	810

[a] Minus sign indicates tension.

[b] 22 yr for columns in air; 17 yr for columns in water.

[c] Yield point.

NOTE.—Column diameter, 5 in. with 1.9 per cent steel and 4½ in. with 5.0 per cent steel. Column length, 48 in. Aggregate, 0 to ½-in. local gravel. Fineness modulus, 4.47. Cement, normal portland, type I. Aggregate-cement ratio, by weight, 8.40 for 2000-psi concrete, 4.80 for 4000-psi concrete. Water-cement ratio, by weight, 0.83 for 2000-psi concrete, 0.53 for 4000-psi concrete. Slump, 6 in. Curing, 21 days moist at 70 F, then stored as indicated. Age at loading, 28 days.

Figure 11 shows the observed length changes for all conditions of loading, reinforcement, and storage. As indicated in other tests, conditions of storage exerted a profound influence on the length changes. While plain concrete columns stored in air shrank up to 1000 millionths, plain concrete columns stored in water swelled 100 millionths. The effect of the steel reinforcement in reducing shrinkage is shown to be in proportion to the amount of steel. While the 22-yr shrinkage of plain concrete was 2½ times the shrinkage at 1 month, the 22-yr shrinkage for columns reinforced with 1.9 per cent steel was only fractionally greater than that at 1 month, and after 1 month no further shrinkage was noted for columns reinforced with 5 per cent steel.

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[6] Building Code Requirements for Reinforced Concrete (ACI 318–56).

The creep curves show trends quite similar to those described for shrinkage. Creep of columns stored in air was about three times that for corresponding columns stored under water. Increasing the percentage of steel reduced the creep in concrete. However, regardless of the condition of storage or of steel percentage, in all cases creep increased for the entire 22-yr period of observation.

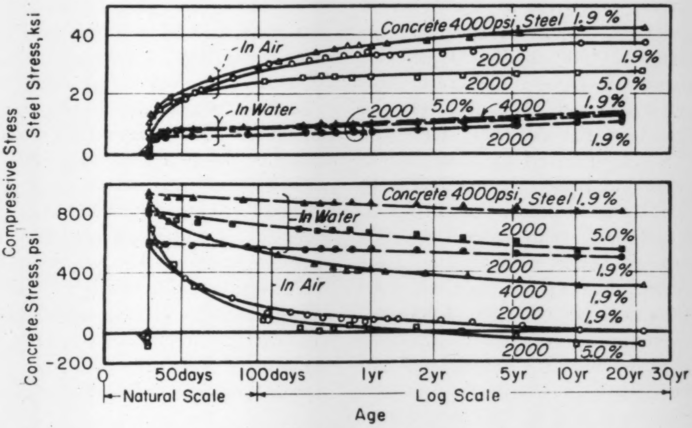

FIG. 12.—Stresses in Steel and Concrete of Reinforced Concrete Columns.
See Table I for column details.

The combined length changes due to creep and shrinkage were five times as large for air storage as for water storage and indicate the strong influence of the percentage of steel on the total length changes. For air storage, length change of the columns containing 1.9 per cent of steel was 55 per cent of that for the plain concrete columns, while for those containing 5 per cent of steel, the length change was only 30 per cent of that for plain concrete. The columns stored under water showed the same general behavior, but with slight changes in the percentages. These effects persisted for the entire period of observation and indicate the value of steel in reducing volume changes.

Average stresses in the steel and concrete were determined from the observed column strains and the complete stress-strain characteristics of the steel. For any strain in the steel, the corresponding average stress was found, which when multiplied by the area of longitudinal steel, gave the portion of the load carried by the steel. The remainder of the total load was carried by the concrete, and the average stress in the concrete was found by dividing concrete load by concrete area.

The stresses in the reinforced columns before and after application of load are given in Table I. The stresses immediately before loading were all referred to the condition existing at the end of the 21-day curing period, when no stresses were assumed to exist. The stresses shown in columns 4 and 5 of Table I were caused by the storage condition during the 7 days prior to loading. For the specimens stored in air, the drying shrinkage of the concrete developed compressive stresses up to 3200 psi in the steel and tensile stresses up to 75 psi in the concrete. For the specimens stored in water, the swelling of the concrete developed tensile stresses up to 700 psi in the steel and compressive stresses up to 15 psi in the concrete.

The stresses immediately after loading are shown in columns 8 and 9 of Table I, and the stresses after long-time loading are shown in columns 10 and 11. For the specimens stored for 22 yr in air, those made of 2000-psi concrete reinforced with 5.0 per cent of steel finally developed a tensile stress of 80 psi in the concrete while the compression in the steel increased from 10,900 to 28,000 psi. For those made of 4000-psi concrete reinforced with 1.9 per cent of steel, the compression in the concrete decreased from 920 to 305 psi and the compression in the steel increased from 10,800 to 42,500 psi, its yield point. For the specimens stored for 17 yr in water, the maximum increase in the compressive stress in the steel while under sustained load was 7000 psi (from 6400 to 13,400 psi) and the maximum decrease in the compressive stress in the concrete was 265 psi (from 820 to 555 psi).

The diagrams of Fig. 12 show the effect of time on the steel and concrete stresses. The steel reinforcement took a gradually increasing share of the compressive load for the first 10 yr, with a corresponding decreasing compressive load on the concrete. After 10 yr the stress changes were insignificant. For the columns stored in air which were made of 2000-psi concrete and contained 5 per cent steel, after 2 yr of loading the shrinkage, when combined with the effects of load, was sufficiently great to produce a net tensile stress in the concrete, which ultimately reached 80 psi. The steel was then carrying the entire load to which the column was subjected plus a compressive load equal to the tension in the concrete.

SUMMARY

For the 4-in. diameter unloaded cylinders and those loaded to 800 psi at 28 days and thereafter stored in air at 50 per cent relative humidity, at 70 F, it was observed that:

1. Creep and shrinkage deformations continued over the entire period of observations, which in some cases was as long as 28 yr; but the rates of change at the later ages were very small.
2. Eighteen to 35 per cent of the total creep after 20 yr occurred in the first 2 weeks of loading, 40 to 70 per cent within 3 months, and 64 to 83 per cent within 1 yr (Fig. 9). The average values were 26, 55, and 76 per cent respectively.
3. Fourteen to 34 per cent of the total shrinkage after 20 yr occurred in the first 2 weeks of drying, 40 to 80 per cent within 3 months, and 66 to 85 per cent within 1 yr (Fig. 10). The average values were 22, 56, and 78 per cent respectively.
4. In many cases the magnitude of shrinkage was practically equal to creep (Figs. 1, 2, 3).
5. Creep increased with a higher water-cement ratio and with a lower aggregate-cement ratio but was not directly proportional to the total water content of the mix (Fig. 1).
6. In these tests, shrinkage was not appreciably affected by either the water content, the water-cement ratio, or the aggregate-cement ratio.
7. Creep of concrete was appreciably greater for type IV (low-heat) than for type I (normal) portland cement (Fig. 2). For type IV cement the creep was greater for the coarse grind than for the fine, but the reverse was true for the type I cement.
8. Shrinkage of concrete was affected by composition and fineness of cement (Fig. 2), being greater for type IV (low-heat) cement than for type I (normal) and greater for the fine-ground cements of both types.

9. Creep of concrete was greatest for crushed sandstone aggregate followed in descending order by basalt, gravel, granite, quartz, and limestone (Fig. 3). The creep for sandstone concrete was more than double that for limestone concrete, showing the importance of materials selection where creep is a factor.

10. Shrinkage was greatest for sandstone concrete, and then in descending order of shrinkage came gravel, basalt, granite, limestone, and quartz concretes (Fig. 3). The shrinkage for sandstone concrete was more than double that for quartz time.

11. The rate of long-time creep of concrete was not appreciably different for $1\frac{1}{2}$-in. aggregate than for $\frac{3}{4}$-in. aggregate, when the paste content was kept constant (Fig. 7).

For ages of loading from 28 to 90 days, stresses from 300 to 1200 psi, storage conditions which ranged from air at 50 per cent relative humidity to immersion in water, and for specimen diameters from 4 to 10 in., the following statements apply:

12. The older the specimen at time of loading, that is, the more complete the hydration of cement, the less the creep (Fig. 4).

13. The creep per unit of stress was only slightly greater at the high stresses than at the low stresses (based on Fig. 4). However, for each increment of higher stress the creep coefficient tends to increase (Fig. 5). (Other tests have shown a very large increase in the creep coefficient as the stress approaches the ultimate strength of the concrete.)

14. The humidity of the surrounding air had a very great effect on both creep and shrinkage (Fig. 6). The shrinkage at 50 per cent relative humidity was about 1.4 times that at 70 per cent relative humidity and about 8 times the swelling for storage in water. The creep in air at 50 per cent relative humidity was about 1.4 times that in air at 70 per cent relative humidity and about 3 times that for storage in water.

15. Creep decreased as the size of specimen increased (Fig. 8), a large part of the difference occurring at a very early age. In general, large specimen size or any other factor which will tend to decrease the movement of internal moisture probably will tend to decrease the creep of concrete.

16. The combined shrinkage or swelling and creep ranged from about 1 to 11 times the instantaneous deformations, the low values occurring for storage in water or fog, for high intensities of stress, and for limestone aggregate; the high values resulted from storage in dry air, at low intensities of stress, and for sandstone aggregate.

From the tests on the reinforced-concrete columns it appears that:

17. Those made of 2000-psi concrete and 5 per cent of steel, which were initially stressed to about 900 psi in the concrete and 9400 psi in the steel and which were stored in air for 22 yr, developed a low tensile stress (80 psi) in the concrete and a high compressive stress (28,000 psi or $1\frac{1}{2}$ times the allowable compressive stress) in the steel as a result of the combined effect of shrinkage and creep.

18. Similarly, for those of 4000-psi concrete with 2 per cent of steel which were initially stressed to about 1000 psi in the concrete and 7600 psi in the steel and which were stored in air for 22 yr, the compression in the concrete was reduced to one third its original value and the stress in the steel was increased to the yield point.

19. For corresponding reinforced concrete columns stored in water for 17 yr, the maximum increase in steel stress with time was only 7000 psi (from 6400 to 13,400 psi) and the maximum decrease in concrete stress was only 265 psi (from 820 to 555 psi).

Acknowledgments:

Many students and staff members of the Engineering Materials Laboratory have been associated with these tests since their inception 30 yr ago. Although it is not possible to give recognition here to all of them, we desire in particular to acknowledge the contributions made by Harmer E. Davis, Professor of Civil Engineering and by Norman E. Haavik and Elwood H. Brown, Research Engineers in the Engineering Materials Laboratory.

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THE EFFECT OF FINE AGGREGATE CHARACTERISTICS OTHER THAN GRADING ON STRENGTH AND DURABILITY OF PORTLAND-CEMENT MORTAR*

By John G. Dempsey[1]

Synopsis

The effect on mortar strength and durability of the characteristics of fine aggregate other than grading is examined through tests of sand-cement mortars made at equal water-cement ratio and containing enough sand so that consistency is the same for all batches.

The investigation is divided into six sections: (1) Mechanical analysis and regrading to the test standard. (2) Classification under the microscope of each size fraction as to edge, surface and shape characteristics; the particle shapes were used to approximate the surface area of the sample. (3) Preparation of mortar samples in accordance with ASTM Method C 87[2] for measuring the mortar making characteristics of sand. (4) Computation of the thickness of the film of water surrounding each solid particle in the mixture and the thickness of the film water and cement slurry surrounding each sand particle. (5) Measurement of tensile and compressive strength, volume change after 28 days of wet curing and a subsequent 28 days shrinkage in laboratory air. (6) Measurement of durability in terms of slow freezing in air and thawing in water and the computation of a "relative durability factor."

The investigation indicated that the cement factor of a cement-sand mortar varies with the roughness, sharpness, and regularity of the surfaces and edges of a natural sand with a fixed gradation, while strength and durability do not necessarily vary proportionally with the amount of cement.

Some question is raised concerning the relative durability of manufactured sand as compared to the natural product. However, the tests confirm the generally held opinion that the durability of a mortar varies inversely with its water requirement, and that the generally accepted shape and surface factors have definite significance in defining sand of superior quality.

Although a great deal of work has been done determining what constitutes a sound fine aggregate material and what kind of a grading curve produces optimum density and workability, comparatively little work has been done on the effect of other characteristics of fine aggregate on strength and durability; particularly with regard to its angularity, type of surface and shape.

The purpose of this paper is to report these characteristics in a group of different sands and consider the results of tests made on mortars in which the gradation of the different sands does not vary.

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[2] Method of Test for Measuring Mortar-Making Properties of Fine Aggregate (C 87 – 52), 1955 Book of ASTM Standards, Part 3, p. 1202.

MATERIALS USED

The cement used was a mixture of equal parts of two brands of type I portland cement purchased in Chicago. At the start of the program, it was mixed together thoroughly and placed in twenty cans with air-tight friction covers so there should be no difference in cements used at different times during the program.

Ottawa sand was used as a control material. Sample No. 110 was a blend of equal parts of Method C 109 and C 190 sands with a fineness modulus of 2.56, and sample No. 111 was C 190 sand (100 per cent passing the No. 20 and retained on the No. 30 sieve). Where referred to for comparative purposes, the average of the two is used.

There are several distinct categories of fine aggregate. The first group consists of samples from three different supply yards of a major Chicago supplier and the yard of each of two others. These were sampled at intervals of several months as a check of the uniformity of a given supply source.

The second series represents the pits of different suppliers in the Chicago area extending from Janesville, Wis., to the Bass River in Michigan.

Two glacial sands were obtained from the state of Washington. The first consists of two natural glacial sands customarily used in a 40 to 60 blend in the city of Spokane. The second is a natural pit-run sand from Stellacoom.

Three samples were received from the island of Puerto Rico. One is very fine, white silica sand from Manati beach. This is the only such material on the island, and while not used for concrete was submitted as being of unique interest. Two other beach sands were submitted, one in common use by residential contractors in the area near Central Constancia and the other a source approved for work on structures at Ramey Air Field. The fourth sample was obtained from the Loiza River, which drains the mountains of the interior.

Four very different samples were received from the state of Missouri. Two of them are manufactured sands: a trap-rock sand having a specific gravity of 3.01 and a rolled flint sand with a specific gravity of 2.59. The natural material consisted of an angular Meramec River sand with a specific gravity of 2.55 and a Missouri sand from deposits near Boonville, with a specific gravity of 2.61.

As a direct comparison of natural and manufactured sand, two samples were received from the Blue Rock quarries in Maine, a glacial deposit. The final group contains four sands from southern California: from Rose Canyon, Mission Valley, Poway (the fine aggregate used for Sutherland Dam), and San Gabriel Canyon.

Each sample as it was received was stored in 2½ gal tight-covered pails until processed. When used in the test mixes it was always recombined to standard grading.

ELIMINATION OF GRADING AS A FACTOR

Following a mechanical analysis using the standard sieve series, the sand was tested in the saturated surface-dry condition for specific gravity, dry rodded weight, dry loose weight, per cent washed through the No. 200 sieve, and for organic matter by means of the colorimetric test.

Immediately after weighing each sieve fraction, a small vial was filled one third to one half full for later examination under the microscope. The individual fractions were then recombined into a

TABLE I.—SAMPLES DEFINED.

Sample	Origin	Specific Gravity	Silt, per cent	Plate Color	Edges, per cent of total		Roughness Factor	Surface Shape, percentage				
					Sharp	Broken		Lamellar	Tabular	Equant	Columnar	Acicular
No. 110	Ottawa	2.67	None	0	...	...	80	...	...	100	...	...
No. 111	Ottawa	2.66	None	0	...	...	80	...	...	100	...	...
Suppliers Group												
No. 116	Yard No. 1	2.66	0.8	1	32	3	100	1	2	95	2	...
No. 121	Yard No. 1	2.65	2.2	1	3	3	96	1	2	94	3	*T*[a]
No. 152	Yard No. 1	2.66	1.2	1	1	7	91	2	6	88	3	1
No. 118	Yard No. 12	2.63	0.6	1	4	*T*	94	*T*	2	98	...	...
No. 123	Yard No. 12	2.67	1.4	1	19	11	95	*T*	2	97	1	...
No. 119	Yard No. 9	2.65	0.5	1	2	...	100	1	...	99	*T*	...
No. 125	Yard No. 9	2.66	1.9	1	10	...	96	...	1	95	4	*T*
No. 117	Lacon	2.65	0.3	2	24	...	96	*T*	*T*	99	*T*	...
No. 122	Lacon	2.63	1.4	1	9	2	96	1	1	97	1	...
Chicago Area												
No. 124	Indiana	2.66	1.4	1	1	2	90	*T*	1	97	2	...
No. 153	Aurora	2.66	0.3	1	...	*NR*[b]	97	2	6	86	6	...
No. 120	Bass River	2.66	2.0	1	...	...	100	...	...	94	6	...
No. 126	Beloit, Wis.	2.65	0.5	1	4	40	100	...	*T*	93	7	...
No. 127	Afton, Wis.	2.65	0.7	1	93	...	100	1	...	96	3	...
No. 128	Janesville	2.66	0.7	1	8	9	94	...	1	93	6	...
No. 129	Fontana, Ind.	2.69	3.2	1	10	3	100	2	5	85	8	...
No. 130	McHenry, Ill.	2.69	1.3	1	12	13	100	3	3	71	22	1
No. 131	Crystal Lake	2.67	1.5	1	13	9	97	*T*	3	92	5	*T*
No. 132	Fox River	2.66	1.5	1	12	10	97	2	4	80	14	*T*
Washington												
No. 135	Spokane	2.65	1.9	1	5	...	102	4	6	77	13	*T*
No. 146	Stellacoom	2.68	1.2	3	7	14	93	2	4	61	31	2
Puerto Rico												
No. 136	Manati	2.61	1.2	1	90	7	86	2	6	84	7	1
No. 137	Constancia	2.60	3.9	2	32	20	98	2	9	75	14	*T*
No. 138	Ramey	2.53	0.3	1	35	23	80	4	13	66	11	6
No. 139A	Loiza River	2.66	1.6	2	49	18	100	3	8	73	16	*T*
Missouri												
No. 140	Meramec River	2.55	1.1	1	35	21	86	...	10	80	9	1
No. 143	Missouri River	2.61	0.2	1	*T*	*T*	81	*T*	3	87	10	...
No. 141	Flint	2.59	0.9	1	92	63	119	20	15	42	20	3
No. 142A	Porphyry	3.01	1.5	1	73	*T*	119	7	11	63	17	2
Maine												
No. 145	Blue Rock	2.67	5.8	1	73	*T*	119	10	10	60	19	1
No. 144A	Blue Rock	2.63	0.6	1	53	37	87	*T*	6	82	12	*T*
California												
No. 148	Rose Canyon	2.65	1.2	2	11	41	107	1	5	70	24	*T*
No. 149	Mission Valley.	2.60	1.5	1	19	34	107	8	6	60	25	1
No. 150	Poway	2.61	1.3	1	29	78	107	2	8	63	27	...
No. 151	San Gabriel	2.61	1.5	1	20	22	102	1	4	65	30	*T*

NOTE: Per cent values are for standardized grading.

[a] *T* = trace.

[b] *NR* = no report.

standardized grading as follows:

Sieve	Per Cent Retained	Accumulated Per Cent Retained
No. 8	00	00
No. 16	20	20
No. 30	20	40
No. 50	48	88
No. 100	10	98
No. 200	2	100
Pan	...	...

Fineness modulus: 2.46.

Repeat samples were taken from four Chicago supply yards at intervals of ten to twelve days as a check on uniformity. These are noted in Table I as the Suppliers Group.

DESCRIPTIVE TERMINOLOGY

The systematic examination of the identifying characteristics of the different samples and their behavior under test required the use of certain descriptive terms. For the purposes of this paper, they are defined as follows:

Particle Edges:

S = Sharp: Refers to particle edges as seen under the microscope

R = Rounded: These edges under the microscope have a complete lack of sharpness so as to continue the texture of one face around to the adjacent one.

B = Broken or serrated: Edges of this character are sufficiently irregular to provide additional keying of the particle into a water-cement matrix.

Surface Roughness:

Under the microscope, the kind of surface of the particle was observed. Subsequently, the average water demand of the different mortar samples containing sand of different roughness was tabulated, and on the basis of this information the roughness factors noted below were set up.

For our purposes, a roughness factor of 100 is defined as a moderately reflective but rough and irregular surface judged to bond well with a cement-water matrix and characteristic of concrete sand requiring about 36 gal per cu yd of mixing water in concrete using coarse aggregate up to 1 in. maximum size.

Kind of Surface Noted	Roughness Factor
Polished	80
Smooth	89
Matte (Dull)	93
Weathered	96[a]
Rough	100
Very rough, fractured	102
Knobby or cavitated and rough	107
Manufactured sand	119

[a] Interpolated value.

Fractured.—Indicates the kind of surface presented by a crystal, broken by impact on a cleavage plane and sufficiently irregular to promote keying as well as bond.

Knobby or cavitated.—Refers to surfaces having either excrescences or cavities which substantially increase area and the keying action of the particle.

Particle Shape:

Five types of particle shapes were recognized and a count made of each type under the microscope:

Lamellar.—Very flat or thin particles in which the thickness is about one tenth of the maximum dimension,

Tabular.—Particles in which the thickness is about three tenths of the maximum dimension,

Equant.—Round or cubic particles in which the ratio of maximum to minimum dimension is close to one to one,

Columnar.—Cylindrical or elongated particles, in which the height is about three times the minimum dimension, and maximum and intermediate dimensions are approximately equal,

Acicular.—Very elongated particles, in which the height is about ten times the minimum dimension, and minimum and intermediate dimensions are approximately equal.

METHOD OF EXAMINATION

After making the mechanical analysis of the original sample, small fractions of the different sieve sizes were examined under a metallographic microscope fitted with the objectives and annular condensers of the modified Leitz dark-field system of illumination. This system emphasizes color, shape, and texture and provides a relatively great depth of focus.

A typical sample was obtained by quartering and requartering each small sample obtained during the mechanical analysis. It was then placed on a slide of black glass. Traverses were made across the sample so as to include not less than 500 individual particles and counts made of each characteristic. The results of the examination are summarized in Table I.

TEST PROGRAM

The test program was confined to mortar mixes. Batches were made in accordance with ASTM Method C 87.[2] In this test, water-cement ratio is maintained at 0.60 by weight of the cement. Consistency is held at a flow of 100 ± 5 per cent when the test sample is dropped through a height of $\frac{1}{2}$ in. ten times in 6 sec on a standard flow table. With the sand always at the same nominal grading and fineness modulus, it was believed that its other characteristics would influence the amount of sand that could be carried in the mixture at standardized consistency. This proved to be the case.

In order to hold the human equation to a minimum, it was decided to mix the mortar mechanically in accordance with ASTM Method C 305[3] instead of manually as required in Section 3 of ASTM Method C 87.

In addition to molding 2-in. cubes, 1 by 1 by 11-in. bars for checking volume change and resistance to freezing and thawing and standard tension test briquets were made for each sample.

In making up the batches, the operator brought the main sample to a saturated surface-dry condition and prepared enough material to make six cubes, six briquets and eight bars.

In every case the basic batch was 1000 g of water, 1667 of the blended type I cement, plus enough of a previously weighed sand at the standardized grading to produce a flow of 100 ± 5 per cent.

Tests for compressive strength were made at 7 and 28 days on a Baldwin-Hamilton testing machine set at the rate of loading specified for small cubes. Tensile strength was measured at the same ages on a Galileo Officine tension testing machine. There is no difference in the human equation between compressive and tensile samples with this type of equipment except what might come from the smaller cross-section of the briquets.

The volume change of the mortar specimens was measured in accordance with ASTM Method C 157.[4] Initial measurement was made as soon as the test bars were taken from the molds. Subsequent measurements were made after 28 days of moist curing and after 3, 7, and 28 days of storage in laboratory air. Moist curing was in water at 73 F.

The test for resistance of the specimens to freezing and thawing was patterned after ASTM Method C 310.[5] Equipment did not permit exact conformity with this test. There was no provision for measuring temperatures at the centers of specimens, the temperature of the thawing cycle was 73 F instead of 50 F, and the length of the thawing cycle was 8 hr. At suitable intervals, the fundamental transverse frequency of the specimens was measured in accordance with ASTM Method C 215.[6] The tests were continued until the specimens had been subjected to 500 cycles or more. In that time none of the test groups showed significant reduction in relative dynamic modulus of elasticity, although one set had spalled so badly that visual inspection suggested failure. There was no serious spalling on the other specimens.

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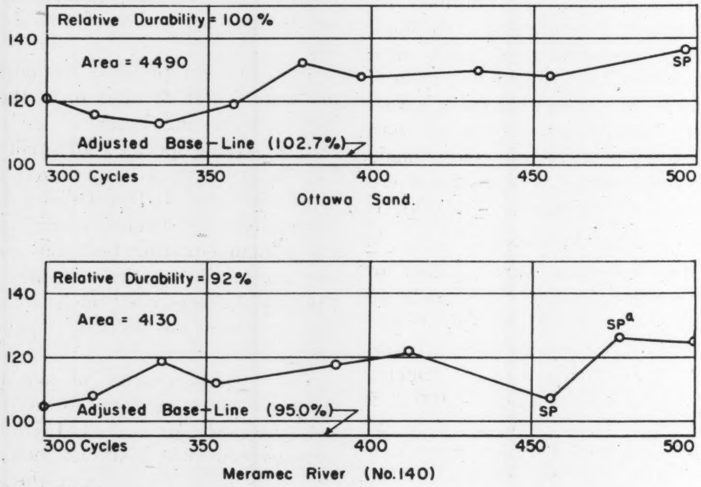
[3] Tentative Method for Mechanical Mixing of Hydraulic Cement Mortars of Plastic Consistency (C 305 – 55 T), 1955 Book of ASTM Standards, Part 3, p. 175.

[4] Tentative Method of Test for Volume Change of Cement Mortar and Concrete (C 157 – 54 T), 1955 Book of ASTM Standards, Part 3, p. 1372.

[5] Tentative Method of Test for Resistance of Concrete Specimens to Slow Freezing in Air and Thawing in Water (C 310 – 56 T), 1956 Supplement to Book of ASTM Standards, Part 3, p. 204.

In view of these conditions, it was decided to measure the area generated by a curve in which per cent of initial dynamic modulus was the ordinate and cycles of freezing and thawing between 300 and 500 was the abscissa. The origin of the curves was, in each case, $x = 300$ cycles and $y =$ original dynamic modulus expressed as 100 per cent. A typical curve is shown in Fig. 1.



[a] Spalling noted.

FIG. 1.—Freezing and Thawing Series as per cent Initial Dynamic "E."

To attain a relative measurement of performance, it was necessary to take account of the variation in the initial value of the dynamic moduli as between individual specimens. This was done by determining the average initial dynamic modulus for the entire series (268 ± 24) and adjusting the base line of the curve (Fig. 1) upward or downward according to the percentage of the individual initial E in relation to the average value for the series. After this was done, the areas beneath the curves were measured and their value recorded as a percentage of the average of the two curves generated by the Ottawa control samples Nos. 110 and 111. The recorded value has been termed "relative durability factor."

COMPUTATIONS

As soon as the exact proportions of the different batches were known, it became possible to study inter-particle relationships. It was thought that an assumption that all aggregate particles were spheres might conceal the effect of variations in surface area or in the thickness of the slurry of water, cement, and air which must be around every larger particle. As a closer approximation of the true unit surface area of the sample, it was assumed that while "equant" particles were spheres, variants were cylinders classified as noted previously under *Particle Shape*. The unit surface areas computed from the observed percentage

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[6] Tentative Method of Test for Fundamental Transverse, Longitudinal and Torsional Frequencies of Concrete Specimens (C 215 – 55 T), 1955 Book of ASTM Standards, Part 3, p. 1355.

TABLE II.—MORTAR BATCHES.

Sample	Origin	Parts Sand	Percentage Flow	Unit Surface Area, sq cm per kg	Slurry Thickness, μ	Water Film, μ
No. 110	Ottawa	3.00	97	47 440	77.4	3.35
No. 111	Ottawa	3.14	105	26 458	110.3	3.40
No. 116	Lockport 1	3.00	105	48 343	66.8	3.32
No. 121	Lockport 1	3.00	97	47 440	64.4	3.34
No. 152	Lockport 1	3.00	101	46 103	66.2	3.32
No. 118	Lockport 12	3.00	103	49 352	63.8	3.31
No. 123	Lockport 12	3.00	102	47 816	63.9	3.31
No. 119	Lockport 9	3.00	97	47 974	63.7	3.32
No. 125	Lockport 9	3.00	103	46 701	65.4	3.31
No. 117	Lacon, Ill.	3.00	97	48 195	63.4	3.31
No. 122	Lacon, Ill.	3.00	101	48 255	63.2	3.31
No. 124	Walcottville	3.00	101	48 492	63.0	3.31
No. 153	Aurora	3.00	100	47 147	64.7	3.31
No. 120	Bass River	3.00	105	47 680	64.1	3.31
No. 126	Beloit	3.06	104	46 524	64.4	3.31
No. 127	Afton	3.00	103	47 519	64.3	3.31
No. 128	Janesville	3.00	105	46 212	66.1	3.32
No. 129	Fontana	3.00	100	45 252	67.4	3.32
No. 130	McHenry	3.00	102	42 449	72.0	3.34
No. 131	Crystal Lake	3.00	98	43 186	70.7	3.33
No. 132	Fox River	3.00	103	44 398	68.8	3.33
No. 135	Spokane	3.00	108	44 662	68.4	3.33
No. 146	Stellacoom	3.16	100	40 698	71.3	3.34
Nos. 136–152	Blend	3.00	106	34 124	89.5	3.38
No. 137	Constancia Beach	2.54	100	46 416	77.7	3.36
No. 138	Ramey Beach	3.00	100	41 335	73.9	3.34
No. 139A	Loiza River	2.51	100	44 708	81.8	3.37
No. 140	Meramec River	3.00	100	47 536	64.3	3.31
No. 143	Missouri River	3.58	105	45 527	56.2	3.27
No. 141	Rolled Flint	2.21	105	43 858	94.6	3.40
No. 142A	Porphyry Chat	3.00	100	39 851	76.7	3.35
No. 145	Blue Rock Quarry	2.31	103	44 438	89.3	3.38
No. 144A	Blue Rock Quarry	3.00	100	45 644	66.9	3.32
No. 148	Rose Canyon	2.46	102	38 592	96.5	3.40
No. 149	Mission Valley	2.43	96	43 274	87.2	3.38
No. 150	Poway	2.38	100	39 076	98.5	3.40
No. 151	San Gabriel	2.64	100	41 827	83.0	3.37

NOTE.—It will be noted that batches Nos. 135 and 136 were made up at flows greater than 105 per cent the established test limit. They could not be repeated because of lack of sufficient material to make up the standard grading.

TABLE III.—TEST RESULTS.

Sample	**Origin**	Strength, psi				Volume Change, per cent						Dynamic Modulus	
		Compressive		Tensile									
		7 days	28 days	7 days	28 days	Damp, 28 days	Air, 3 days	Air, 7 days	Air, 28 days	Total Shrinkage	Total Volume Change	Initial *E*	Relative Durability, per cent
No. 110.......	Ottawa	2280	3380	325	425	+004	031	020	026	073	077	264	80
No. 111.......	Ottawa	2140	3760	320	460	+016	044	025	032	085	101	258	120
No. 116.......	Lockport 1	3020	3680	440	525	+013	*NR*[a]	*NR*	*NR*	*NR*	*NR*	252	91
No. 121.......	Lockport 1	2980	5140	420	580	+010	019	023	044	077	086	273	118
No. 152.......	Lockport 1	3720	5260	400	535	+016	043	020	023	070	086	352	153
No. 118.......	Lockport 12	2600	4590	410	520	+020	048	020	037	084	105	257	*NR*
No. 123.......	Lockport 12	3090	4580	380	570	+006	028	014	039	075	081	284	*NR*
No. 119.......	Lockport 9	3060	5450	420	520	*NR*	*NR*	*NR*	*NR*	*NR*	*NR*	271	127
No. 125.......	Lockport 9	3620	5930	455	580	+018	025	028	070	107	123	294	116
No. 117.......	Lacon	2370	5080	380	510	+011	046	025	040	100	111	237	83
No. 122.......	Lacon	2260	4690	370	520	+005	021	020	044	079	085	281	111
No. 124.......	Indiana	3210	4890	370	520	+010	026	015	049	080	090	290	*NR*
No. 153.......	Aurora	3025	4630	448	536	+014	034	024	028	072	086	280	115
No. 120.......	Bass River	2590	4670	375	505	+039	072	010	033	076	115	310	133
No. 126.......	Beloit	3360	5270	410	555	+014	022	021	046	075	089	324	75
No. 127.......	Afton	2550	4380	375	510	+022	036	031	049	094	116	277	113
No. 128.......	Janesville	2790	4920	450	560	+027	028	024	039	064	091	301	135
No. 129.......	Fontana	3110	5190	365	405	+005	018	019	049	081	086	272	134
No. 130.......	McHenry	3100	5130	405	460	+001	012	015	047	074	074	289	147
No. 131.......	Crystal Lake	2560	4990	350	480	+018	035	013	053	083	101	288	107
No. 132.......	Fox River	2500	5270	370	510	*NR*	*NR*	*NR*	*NR*	*NR*	*NR*	287	123
					540	+076							
No. 135.......	Spokane	2900	5090	445			109	035	033	101	177	292	102
No. 146.......	Stellacoom	2725	4180	340	480	+018	025	041	030	078	096	274	103
Nos. 136–152..	Blend	2870	5000	375	515	+016	024	038	023	069	085	292	126
No. 137.......	Constancia	3080	4390	435	565	+086	045	035	057	051	137	244	78
No. 138.......	Ramey	2860	4150	375	530	+018	022	021	037	063	080	273	118
No. 139A.....	Loiza	*NR*	5010	*NR*	540	+024	061	010	025	072	096	263	106
No. 140.......	Meramec River	2580	4520	340	420	+020	016	028	044	068	088	283	92
No. 143.......	Missouri River	2690	4710	380	460	+022	022	024	021	045	067	298	137
No. 141.......	Rolled Flint	3230	5880	485	685	+022	025	020	035	058	080	277	78
No. 142A.....	Porphyry Chat	2980	4840	*NR*	630	+009	022	026	037	076	085	292	58
No. 145.......	Maine	2830	4750	415	580	+022	031	034	052	095	117	234	98
No. 144A.....	Maine	3040	4420	425	555	+018	024	036	034	076	094	280	97
No. 148......	Rose Canyon	3225	5190	375	490	+024	053	017	038	085	108	232	46
No. 149.......	Mission Valley	3190	5330	455	590	+022	058	017	040	093	115	251	62
No. 150.......	Poway	3275	4800	385	505	+023	*NR*	*NR*	*NR*	074	097	250	76
No. 151.......	San Gabriel	3135	4865	445	530	+018	026	026	056	091	108	257	104

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[a] No report.

of different shapes are listed in Table II.

The specific gravity of the cement used was 3.16, and its unit surface area 1670 sq cm per g. The thickness of the water film which must surround every solid particle is the total volume of water divided by the sum of the surface areas of the cement and sand in the batch (Table II).

In calculating the thickness of the slurry of cement and water surrounding a sand particle, it is assumed that no air is included. It is computed by dividing the total absolute volume of water and cement by the surface area of the sand in the batch (Table II).

COMPARISON OF BEHAVIOR

It is natural to compare behavior characteristics according to the origin of the different samples. This has little significance here, as the samples were chosen for differences rather than similarities, or points deemed of special interest by the supplier. The only exception is the Chicago area. To check the reliability of results, a deliberate attempt was made in the case of certain suppliers to obtain similarity rather than difference. Since they were easily accessible, 12 samples were also obtained from the area at large to see how much significant difference there might be in an area thought to be similar in its geological history. These varied so little from one another that they were considered as one material and their average is so used in this summary.

Comparing results of tests on the basis of the observed sand characteristics, it was noted that smooth and even polished surfaces are not as detrimental as is generally believed. A detailed summary follows.

Edge Characteristics:

Rounded.—Sand with rounded edges includes the Ottawa control samples. The average 28-day compressive strength of all the rounded types was 4560 psi with a coefficient of variation of 14 per cent. Exclusive of the Ottawa control mix, minimum strength was 4180, maximum 5260 psi. Relative durability after 500 cycles of freezing and thawing averaged 119 per cent with a coefficient of variation of 18 per cent. Minimum was the Ottawa control series, set by definition as 100 per cent.

Sharp.—The natural sand with the sharpest edges is sample No. 144 from the state of Maine. Generally, the sands with sharp edges made mortar with a minimum compressive strength of 4390 psi and a maximum of 5010 psi. The relative durability with respect to Ottawa control sand was below 100, averaging 93 per cent with a coefficient of variation of 11 per cent. Average shrinkage was 0.102 per cent, similar to sand with rounded edges. The average shrinkage was increased by the Constancia sand (No. 137), which was 0.137 per cent. This sand also had the lowest durability in the group—78 per cent.

Manufactured.—As would be expected, the manufactured sand all had extremely sharp-edged particles. Relative durability was 78. The porphyry chat (No. 142A) was reported on visual inspection to have spalled practically to destruction, with relative durability 58. The rolled flint (No. 141) showed the same durability as the average: namely, 78. The total volume change of the manufactured sands investigated was moderate, showing an average of 0.094 per cent. The outstanding characteristics of the three materials were their very high roughness factor, extremely sharp particle edges, and relative durability below 100.

Roughness Factor:

Smooth.—In the scale adopted for this inquiry, smooth to polished surfaces represent a minimum roughness factor. Samples with this type of surface included the Ottawa control sands and showed an average 28-day compressive strength of 4460 psi. Without the control, the minimum was 4200 psi and the maximum 5000 psi. Coefficient of variation was 11 per cent. Relative durability was 113 per cent with a coefficient of variation of 22 per cent. The average total volume change was 0.080 per cent, with a coefficient of variation of 9 per cent. The samples varied widely in surface area but showed low shrinkage, good durability and satisfactory strength. Sample No. 143 from the Missouri River near the city of Boonville was a sand in which many of the particles had been brought to a high polish.

Rough.—The roughest natural surfaces include the Chicago area sands and two of the California samples. Compressive strength at 28 days was over 5000 psi in all cases. However, average relative durability was 83 per cent, influenced by the California values of 46 and 62 per cent, respectively. Shrinkage was an average of 0.124 per cent. Minimum was 0.095 and maximum 0.177 per cent.

Manufactured.—Surfaces of manufactured sand were fine-grained to rough and fractured, varying in the same order as volume change. Durability and strength had no inter-relationship. Maximum strength was obtained using the sharpest aggregate (No. 141).

PARTICLE SHAPE

The five samples showing a minimum percentage of lamellar pieces had an average compressive strength of 4600 psi in 28 days. Except for the Ottawa control sands, minimum was 4700 psi and maximum 5160 psi. Average relative durability was 109 per cent of Ottawa sands with a minimum of 92 and a maximum of 137 per cent.

Average maximum volume change was 0.083 per cent with a minimum of 0.067 and a maximum of 0.095 per cent.

Natural sand containing a maximum number of lamellar particles had a slightly increased compressive strength of 4860 psi in 28 days. Average durability was 91 per cent with a minimum of 62 and a maximum of 108. Total volume change averaged 0.124 per cent with a minimum of 0.080 and a maximum of 0.177 per cent.

Manufactured sand contained still higher percentage of lamellar particles and stacked up as noted previously, that is, good strength but relative durability below 100 per cent of the Ottawa control sands.

Tabular Shapes:

Sands having a minimum percentage of tabular particles averaged 4500 psi in 28 days and exhibited a uniformly superior durability, averaging 114 per cent. Total volume change was also moderate, although the group contained one California sample with a value of 0.108 per cent. The average was 0.089 per cent; minimum, 0.067; maximum 0.108 per cent. It is noted that the durability of all samples was over 100 per cent of the Ottawa control sand even though total volume change was quite variable.

Sands with a maximum percentage of tabular particles showed a high variation with respect to durability and strength in spite of very uniform volume changes. The maximum number of tabular particles was counted in sample No. 138. Average 28-day compressive strength was 4920 psi with a minimum of 4130 psi and a maximum of 5880 psi. Average relative durability was 84 per cent with a minimum of 58 and a maximum of 108 per cent. Total volume change was 0.083 per cent with a minimum of 0.080 and a maximum of 0.088 per cent.

For manufactured sand, maximum and minimum were the rolled flint (sample No. 141) and porphyry chat (sample No. 142A) respectively.

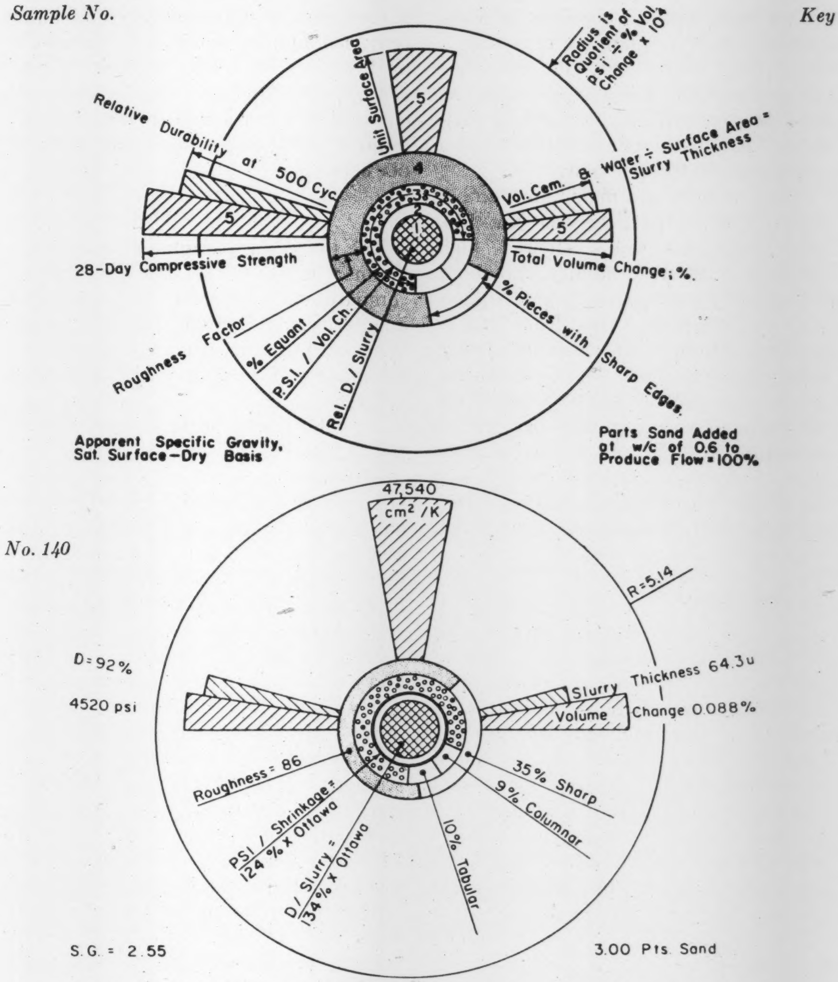
Sample No.
Key
Radius is Quotient of p.s.i. ÷ % Vol. Change x 10⁴
Unit Surface Area
Relative Durability at 500 Cyc.
Vol. Cem. & Water ÷ Surface Area = Slurry Thickness
5
4
3
2
1
5
5
28-Day Compressive Strength
Total Volume Change; %.
% Pieces with Sharp Edges.
Roughness Factor
% Equant
P.S.I. / Vol. Ch.
Rel. D. / Slurry
Apparent Specific Gravity, Sat. Surface-Dry Basis
Parts Sand Added at w/c of 0.6 to Produce Flow = 100%
No. 140
47,540 cm² /K
R=5.14
D=92%
4520 psi
Slurry Thickness 64.3u
Volume Change 0.088%
Roughness = 86
35% Sharp
9% Columnar
PSI / Shrinkage = 124 % x Ottawa
D / Slurry = 134% x Ottawa
10% Tabular
S.G. = 2.55
3.00 Pts. Sand

FIG. 2.—Plan for Summarizing Tests and Observations and Typical Summary Sheet.

Columnar Particles:

The group having a minimum number of columnar particles exhibited moderate volume change and high durability. The percentage of particles with sharp edges ranged from 1 to 40 per cent and the number of equant particles from 73 to 96 per cent. Average 28-day compressive strength was 4760 psi, with a coefficient of variation of 13 per cent. The Ottawa control sand was 3570 psi, and the next lowest with respect to columnar pieces, 4825 psi. Maximum strength was 5260 psi. The average relative durability was

119 per cent with a coefficient of variation of ±0.23, a minimum of 92 and a maximum of 153 per cent. Volume change was very uniform. The average value was 0.086 per cent with a coefficient of variation of 0.06, a minimum of 0.078 and a maximum of 0.095 per cent.

The sand having a maximum number of this type of particle was No. 146. This group as a whole showed high shrinkage, variable durability, and about 60 per cent of the particles were equant in shape. Sharpness varied from 7 to 29 per cent. Strength and volume change were quite uniform. Average 28-day compressive strength was 4800 psi with a coefficient of variation of 8 per cent, a minimum of 4180, and a maximum of 5330 psi. Average relative durability was 86 per cent with a coefficient of variation of 21 per cent, a minimum of 62 and a maximum of 104 per cent.

Acicular Pieces:

Five of the samples investigated showed zero or traces only of this type of particle. Only with respect to compressive strength was there any indication of uniformity. Average 28-day strength was 4590 psi with a coefficient of variation of 12 per cent. Minimum except for Ottawa control sand was 4200 and maximum 5160 psi. The average durability was 106 per cent with a minimum of 76 and a maximum of 137 per cent. Average total volume change was 0.085 per cent with a minimum of 0.067 and a maximum of 0.097 per cent.

The sands with a maximum percentage of these stick-shaped pieces included two manufactured sands. The highest proportion was counted in sample No. 138. Although the other test results show wide variations, volume change is quite uniform. The average compressive strength was 4760 psi with a coefficient of variation of 15 per cent, with a minimum of 4130 and a maximum of 5880 psi. Average durability was 87 per cent with a coefficient of variation of ±0.20 per cent, a minimum of 58 and a maximum of 108 per cent. The average volume change was 0.085 per cent with a coefficient of variation of ±0.06, a minimum of 0.080 and a maximum of 0.095 per cent.

SURFACE AREA

The preceding paragraphs review the manner in which the different samples compare with respect to roughness, angularity, and individual particle shape. When surface area is compared, there is not much uniformity in the quality of the different mortars, so far as these tests show. The lowest surface areas include the Ottawa control sand.

Minimum Area. — Compressive strength was fairly uniform with a coefficient of variation of 13 per cent. The average was 4780 psi. Average relative durability was 82 per cent with a minimum of 46 and a maximum of 126 per cent. Average total volume change was 0.097 per cent with a minimum of 0.078 and a maximum of 0.115 per cent.

Maximum Area.—Samples with maximum surface area show a similar lack of unformity except with respect to strength. The average compressive strength was 4910 psi with a coefficient of variation of 7 per cent, a minimum of 4390 and a maximum of 5260 psi. The average relative durability was 111 per cent with a minimum of 78 and a maximum of 153 per cent. The average volume change was 0.101 per cent with a minimum of 0.086 and a maximum of 0.137 per cent.

Graphs were made for each sample summarizing observations and test results. Referring to the key (Fig. 2(*a*)), the diameter of the center (*1*) is proportional to the relation of the fraction relative durability divided by slurry thickness to a similar value for the Ottawa control. In the same way the fraction strength divided by volume change is plotted (*2*). A following concentric circle of arbitrary diameter (*3*) forms a ring which is divided into segments proportional to the per cent equant, columnar and tabular particles in the sample. At an additional radial increment proportional to the numerical roughness factor (*4*) a circle is drawn. The ring formed has an unshaded portion proportional to the per cent of sharp edges observed. Radial sectors (*5*) are drawn with radii proportional to the values found for 28-day strength, relative durability factor "D," unit surface area, slurry thickness and volume change. The large outer circle emphasizes strength divided by volume change in the manner shown.

TABLE IV.—SUMMARY GROUP.

Item	Average	Coefficient of Variation, per cent
MORTAR TESTS		
Parts by weight for flow of 100 per cent	3.01	9
Compressive strength, psi	4810	7
Durability, per cent	118	18
Total volume change, per cent	0.086	4
Estimated cement factor for concrete, sacks per cu yd	5.03	7
ROUGHNESS, ANGULARITY AND SHAPE		
Roughness factor	93	13
Broken edges, per cent	15.3	35
Surface shape, per cent:		
Lamellar	1.4	100
Tabular	6.9	56
Equant	82.1	10
Acicular	1.0	198
Surface area, sq cm per kg	44.060	10

Figure 2 (*b*) shows the appearance of one of the completed graphs. The completed series made a set of data readily classified and examined. All graphs showing low volume change with superior durability and strength were separated from the series. The result was a group of samples showing quite good uniformity except with respect to edge sharpness. This group points to the characteristics of sand that result in superior mortars with respect to durability, strength, and volume change.

The average value and uniformity as expressed by the coefficient of variation of the performance and identifying characteristics of this group are shown in Table IV. A consideration of the limits of uncertainty and the control ranges derived from Table IV suggests that the general characteristics and response to the mortar-making test of a superior sand should be as follows:

GENERAL CHARACTERISTICS.

Unit surface area, sq cm per kg	38 280	to	47 780
Roughness factor	84	to	104
Broken edges, per cent of total	11.2	to	25.8
Equant shaped pieces, per cent	65	to	90
Lamellar, per cent	0	to	2.0
Tabular, per cent	4.3	to	8.3
Acicular, per cent	0	to	2.4

MORTAR MAKING TEST AT WATER-CEMENT RATIO OF 0.60

Parts by weight for flow of 100 per cent	2.72	to	3.36
28-day compressive strength, psi	4300	to	5100
Total volume change, per cent	0.77	to	0.97
Durability relative to Ottawa control sand, per cent	95	to	125

MANUFACTURED SANDS

The three manufactured sands at standardized grading in the mortar-making test required more cement and water than natural sand to produce a given consistency, confirming that concrete made with it must be high in cement and consequently relatively expensive.

The samples from the Blue Rock quarry (Nos. 144A and 145) show the effect of crushing. The two are from the same glacial deposit, one natural and the other manufactured. The roughness factor changed from 0.92 to 1.19, the percentage of sharp-edged pieces from 53 to 73, and the percentage lamellar pieces from a trace to 10½ per cent, tabular pieces from 6 to 10 per cent, and columnar pieces from 12 to 19 per cent. Acicular or stick-shaped pieces jumped from 0 to 1.2 per cent and surface area decreased from 45,600 to 44,400 sq cm per kg. The per cent cement in total solids increased from 21.8 to 26.9.

Even though total volume change increased from 0.088 per cent for the natural sand (sample No. 144A) to 0.117 per cent for the other, there was little or no change in relative durability between the two: 97 per cent for the natural sand and 98 per cent for the manufactured sand. Further, in these two samples, derived from the same deposit, there was no significant difference in the 28-day compressive strength. This confirms what common sense has long assumed; namely, intrinsic capacity for high durability and strength resides in the native parent material when processing is appropriate to the intended use.

The other two manufactured sands show moderate volume-change, relative durability of 78 per cent for the rolled flint sand and 58 per cent for the porphyry chat. On visual inspection, the porphyry chat had spalled so badly at the end of the test run that visual inspection indicated failure. There was no visible damage to the other samples. Whether this relative drop in durability would be characteristic between the three samples under more rapid and severe exposure to freezing and thawing is not known.

CONCLUSIONS

1. The manufactured sands subjected to the mortar making test in this program required more cement and water than the natural sands at equal water-cement ratio for equal consistency and showed a relative durability below that of the Ottawa control mixes.

2. Smooth and even highly polished surfaces do not reduce strength and durability so far as these tests indicate.

3. There was no correlation between high or low surface area and good test results except that when combined with regular shapes and smooth surfaces, an increase in surface area increased the economy of the resultant mix. At the standardized grading used, a satisfactory surface area lies between 40,000 and 48,000 sq cm per kg. A test mortar prepared with it at a water-cement ratio of 0.60 should require from 2.70 to 3.30 parts of sand by weight of the cement to produce a flow of 100 per cent in the mortar-making test.

4. This investigation suggests that the qualities of surface, edge and shape in a superior sand are as follows:

Roughness factor	0.89 to 1.00 (fine grained to rough)
Equant shaped pieces	Above 70 per cent
Lamellar shapes (thin plates)	Not over 2 per cent
Tabular pieces	Not over 12 per cent
Acicular pieces	Not over 3 per cent

Acknowledgment:

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Engineer of Materials, Missouri State Highway Commission; Francisco Rexach, Manager, National Readymix Inc., Santurce, Puerto Rico and Charles F. Parker, Chief Engineer, W. H. Hinman, Inc., Portland, Me., through whose interest and cooperation the samples for the program were obtained.

COLLATERAL INFORMATION

(1) 1955 Book of ASTM Standards, Am. Soc. Testing Mats., Part 3 (1955).

(2) Method of Test for Flat and Elongated Particles in Coarse Aggregate (Designation CRD-C 119-53), Concrete Research Div., Waterways Experiment Station, Corps of Engineers, Jackson, Miss.

(3) Method of Test for Particle Shape of Fine Aggregate (Designation CRD-C 120-48), Concrete Research Div., Waterways Experiment Station, Corps of Engineers, Jackson, Miss.

(4) Bryant Mather, "Test of Fine Aggregate for Organic Impurities and Compressive Strength in Mortars," ASTM BULLETIN, No. 178, Dec. 1951, p. 35 (TP229).

(5) Recommended Practice for Petrographic Examination of Aggregates for Concrete (C 295 - 54), 1955 Book of ASTM Standards, Part 3, p. 1248.

DISCUSSION

MR. J. E. GRAY[1] (*presented in written form*).—Manufactured sand or stone sand for use as a fine aggregate in portland-cement concrete is a special product of many crushed stone operations. Probably the greatest impetus to the use of manufactured sand has come from the Corps of Engineers which has used stone sand in such dams as Wolf Creek, Allatoona, Buggs Island, and many others. The anticipated life of these dams is probably greater than that of any other type of concrete structure, which surely indicates that stone sand is considered to be a satisfactory and durable fine aggregate.

It may be well to consider just what is manufactured sand or stone sand. Screenings is a finely graded end product of the crushing operation which essentially consists of all material passing the $\frac{3}{8}$ in. or No. 4 sieve and contains all the dust of fracture with no control on gradation. In general, screenings have a high percentage between the Nos. 4 and 30 sieves, a low amount between Nos. 30 and 100 sieves, a high dust content, and unless further processed is often an unsatisfactory fine aggregate. Stone sand, on the other hand, is a specially prepared product for which screenings may be used as a parent material or coarser stone may be used in specially designed mills for production of sand. The product has a controlled gradation including dust content, and in general the milling gives an improved shape of particle over that of a straight crushed material.

The commercial production of stone sand is increasing as the supply of natural sand becomes scarce and expensive. Costwise, stone sand cannot compete with locally available natural sand of good quality because, aside from the normal cost of drilling, blasting, crushing, and screening, a large capital investment is required for the processing of stone sand. Therefore, stone sand has been developed in those areas where natural sand is not readily available, as in sections of the states of Virginia, West Virginia, Tennessee, and Ohio. Recently considerable production has started in Maryland. In these areas the parent rock is limestone or dolomite. The Radford Dam in Virginia and the Henley Bridge in Knoxville, Tenn., are notable examples of excellently performing concrete in which the fine aggregate was commercially produced stone sand.

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[1] Engineering Director, National Crushed Stone Assn., Washington, D. C.

It is extremely questionable whether any of the three manufactured sands used in the investigation reported upon by Mr. Dempsey are representative of the stone sand commercially manufactured for use as fine aggregate. Two are from Missouri whose Highway Department uses no stone sand since Missouri has an abundant supply of natural sand.[2] One of these is a "rolled flint" and the other was a traprock or porphyry of high specific gravity. The "rolled flint" and "porphyry chat" are apparently both waste products from the zinc mines. The third stone sand was produced from a glacial deposit from Maine which "showed the effect of crushing," for the other sample from Maine was a natural sand from the same deposit. While the author makes no claim that the manufactured sands are representative of commercially produced stone sand, one would in all reason expect that such an investigation would include at least one manufactured sand of known good quality from an established and accepted source.

Particle shape was studied by means of the microscope on separations into five shape classifications. These must have been quite laborious to determine, have a high personal equation, and difficult to reproduce to the indicated precision. Moreover, this shape classification is impractical in that the method does not lend itself to application for specification purposes. A rather simple practical method has been developed for controlling particle shape to assure satisfactory workability that is reproducible and is used in specifications. The method is based on the fact that when sand is separated into its different size fractions, such as the No. 8 to No. 16, No. 16 to No. 30, and No. 30 to No. 50, the more elongated and flatter the particle shapes, the higher will be the percentage of voids in these different size fractions. A. T. Goldbeck (**1**)[3] described the method of test essentially as follows:

> "The method consists of sieving the sand into these three sizes and testing each fraction for its voids as follows: 'The apparatus consists of a small cylinder $2\frac{7}{8}$ in. in diameter and $5\frac{1}{2}$ in. high; a truncated cone-shaped funnel having a large diameter of $5\frac{1}{2}$ in. and a small diameter of 1 in. is supported over the center of a cylinder so that the bottom of the funnel is 1 in. above the top of the cylinder. The sample is placed in the funnel and, after filling, the sand is allowed to flow freely into the cylinder to overflowing. Without jarring, the excess is carefully struck off from the cylinder and the weight of the sand remaining is determined. The average of three determinations is used to calculate the voids."

Table V shows typical results of voids determined as described above.

Certainly the evidence is clearly in favor of a low void content indicative of a cubical particle shape for producing good workability in concrete. It has been the suggestion by the National Crushed Stone Assn. that the void determination should not exceed 53 per cent in order to assure material of reasonably satisfactory particle shape.

The Virginia Highway Department has a specification requirement for maximum percentage of voids to control the shape of particle and uses essentially the above described method of test. The Ohio Highway Department controls shape of particle of stone sand by requiring that not more than a stated percentage of the

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[2] Highway Research Board Circular 363.

[3] **The boldface numbers in parentheses refer to the list of references appended to this discussion, see p. 1142.**

TABLE V.—SHAPE OF PARTICLE (EXPRESSED AS PER CENT VOIDS).

Laboratory Number	Preparation	Specific Gravity	Average Voids, per cent	Concrete Workability
35.........	Laboratory gyratory crusher	2.77	58.2	...
1766.........	Unprocessed screenings	2.83	56.5	Very poor
1771.........	Unprocessed screenings	2.98	54.3	Very poor
2343.........	Rod mill and ring mill	...	50.3	Excellent
2421.........	Hammer mill, no grates	...	50.9	Excellent
2674.........	Short head cone and rolls	...	51.7	Good
2671.........	Vertical ring roll	...	49.4	Excellent
577.........	Commercial stone sand	2.72	51.2	...
633.........	Commercial stone sand	2.71	52.2	...
1769.........	Commercial stone sand	2.73	51.7	...

No. 8 to No. 16 material shall pass a slotted or oblong opening of specified dimensions.

A great deal of work is represented by this paper to reconfirm what have been rather well established characteristics of fine aggregate for portland-cement mortar, namely, angular particles require more water for a given consistency than rounded particles. In 1938, Goldbeck (2) reported "the more angular the sand, the higher must be the water content for a given consistency. With the effect of gradation eliminated by holding it constant for all sands, the primary purpose is to study the effect of shape of particle on the properties of mortars."

The author's test program was confined to mortar mixes made in accordance with ASTM Method C 87. However, 1 by 1 by 11-in. non-air-entrained mortar bars were tested for durability by a freezing-and-thawing test. The author states "the three manufactured sands of standardized grading in the mortar-making test required more cement and water than natural sand to produce a given consistency, confirming that concrete made with it must be high in cement and consequently relatively expensive." There are no data in this investigation to warrant a statement on the relative cost of concrete and, furthermore, the implication is not in accord with experience.

The relative concrete-making properties of two fine aggregates should be determined by testing concrete made with normally graded materials as produced in a given locality. The following data show the type of information obtained by making comparative concrete tests:

PROJECT 49–13

Coarse Aggregate................ Fine Aggregate..................	1-in. Stone Natural Sand	1-in. Stone Stone Sand
Non-Air Entrained Structural Concrete		
Cement factor, sack per cu yd..................	4.89	4.91
Water content, gal per cu yd..................	39.9	39.0
Slump, in..................	4.75	4.25
Compressive strength, 28-day, psi..................	2130	2540
Durability,[a] (cycles to failure..................	4	7
Air Entrained Structural Concrete		
Cement factor, sack per cu yd..................	4.92	4.91
Water content, gal per cu yd..................	36.0	33.8
Slump, in..................	5.5	4.75
Compressive strength, 28-day, psi..................	2020	2610
Durability,[a] 80 cycles......	no failure	no failure

[a] In the slow freezing-and-thawing test, the non-air-entrained concrete exhibited mortar failure very rapidly. With air entrainment the stone sand fine aggregate was fully as durable as the natural sand when incorporated into concrete.

It is indicated that concrete made with sound aggregates may be made durable with the use of entrained air and with no increase in cement factor. Concrete tests offer the most direct approach to the evaluation of the concrete-making properties of stone sand. Other tests, such as those made on mortars, with artificially graded samples of sand and without the use of entrained air, may give wrong indications of the actual behavior and economic value of stone sand as it is used in concrete.

MR. J. G. DEMPSEY (*author*).—All materials submitted, manufactured or otherwise, were materials in accepted use with one exception, which was not touched on. We were interested in getting samples with as wide a variation in their particle characteristics as we could. We were not particularly interested in whether they were manufactured or natural sands. The picture of the manufactured sand was somewhat distorted because I did not give you data on the two sands that came from the same source.

These two sands came from the State of Maine. The natural sand had a shrinkage of 0.081 per cent; a durability factor of 99; and a strength of 4980 psi in 28 days. The manufactured sand from the same quarry had 2.31 parts of sand, instead of 3, for a flow of 100 per cent. It had a shrinkage of 0.117 per cent; a durability factor of 98, practically the same; and a strength of 4750 psi, in 28 days. I do not consider the difference of 200 lb in that single test particularly significant. But the fact that the two sands from the same parental deposit showed a similar basic durability is of interest.

I wish to emphasize that we were looking for widely varying kinds of sand and not necessarily sands representing one particular class or area.

MR. C. C. CONNOR[4] (*presented in written form*).—Time has not been available for a complete study of the author's data, but there is no question about their importance. He mixed many different sands, all having a standard grading, with a constant amount of a standardized cement paste. The mortars were mixed to a flow of 100 ± 5, and the properties of the mortars were thus dependent almost entirely upon the characteristics of the sand other than grading. The variation in values he obtained from the various mortars was truly astonishing.

Compressive strength at 28 days varied from 3680 psi to 5930, and the sand-carrying capacity of the paste varied with the different sands from 2.21 parts by weight to 3.8 parts. The durability of the mortars under freezing-and-thawing tests varied from 46 up to 153, according to the author's scale, and the total volume change varied from 0.067 per cent to 0.177 per cent. There was a considerable tendency for the poor values to scatter. Using a rather arbitrary scale, I would judge that 14 of the 33 mortars were unsatisfactory. Seven of these were unacceptable because of a single poor value.

The variations were large and are important to both concrete and masonry mortar. The reasons for these wide variations should be established if possible, and limits established. The author has suggested limitations on the surface characteristics and the percentages of particle shapes which appeared to be the more influential. These limitations are very helpful, but they permitted five sands to pass that I regard as undesirable and rejected four that were satisfactory.

To better determine how the characteristics of the sand influenced the properties of the mortar, I rearranged the author's data to show the progressive changes in compressive strength, the carrying capacity of the paste for the various sands, the durability and the total volume change in the mortars.

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[4] Consulting Engineer, Verona, N. J.

There was no clear-cut evidence that any sand characteristics studied, or any obvious combination of those characteristics was responsible for the variation in compressive strength. With mortars having equal parts of sand by weight, there were some indications that strength increased with the roughness factor and decreased with an increase in the percentage of particles with sharp edges; but the evidence was not conclusive. There were too many exceptions.

The lack of any consistent relation between the compressive strength of the mortars and the characteristics of the sand seemed to indicate one or more hidden factors. The geological origin of the sands was a possibility. It was assumed that the sands from each of the different Lockport yards were similar geologically and that those from Lacon were likewise similar. The sands from Maine were known to be from the same source. The sands from the same location produced mortars with much similar compressive strengths, except for sand No. 116 which dropped over 1400 psi below its companion sands. Sand No. 116 was considerably higher in its percentage of particles with sharp edges. There was a tendency for mortars with sands of a similar geological origin to vary more in durability and volume change than in compressive strength. It looked as though geological origin was not the only hidden factor. It should be noted that the use of parts of sand by weight tended to cover up the variation in parts of sand by volume. This might have had an effect.

I would like to ask the author if my assumptions concerning the common geological origin of sands from the same locations have been correct.

In regard to the effect of the studied characteristics of sand on the sand-carrying capacity of the standard paste, the indications were quite definite. Roughness seemed to be the major factor; but it required a combination of a roughness factor above 97, a total percentage of particles with sharp and broken edges over 40, and equant shapes below 75 per cent to increase the resistance of the sand to flow until, at the flow required, the paste could not carry as much as three parts by weight. The parts of sand by weight carried by the paste varied from 2.21 to 3.58 in the various mortars.

Here, also, the use of parts of sand by weight clouded the picture considerably. It is not the weight of a sand that affects the flow of the mortar but rather the sand's volume, the volume of its voids, and its other characteristics.

The durability of the mortars in freezing-and-thawing tests varied from 46 up to 153 as measured by the author's scale. There was a strong tendency for durability to be reduced by the same combination of characteristics of sand that reduced the carrying capacity of the paste. This combination consisted of a high roughness factor, a high total percentage for sharp and broken edges, and a low percentage of equant shapes. There were exceptions—too many to be comfortable. Geological origin probably affected durability but did not seem to explain all the exceptions to this formula. The fact that one sand that produced mortar of low durability had been used in a big western dam emphasizes the need to determine the cause of the effect of sand on this property.

The total volume change in the mortars covered a wide range, varying from 0.067 to 0.177 per cent. This variation, caused entirely by the sand, is more than 1 in. in 100 ft and is more than sufficient to explain the cracks that occur in the best of concrete. It may well have been a major cause of the cracks in basement concrete, which is a problem I am now studying.

There was no combination of the sand characteristics the author studied that showed any reasonably consistent relation to the total volume change. The values for volume change scattered without any obvious pattern. This condition strongly suggests one or more factors which are unknown or were not considered.

Geological origin of aggregate affects volume change, but there was considerable variation in the total volume change of mortars with sands from similar locations, indicating the influence of some factor other than geological origin.

Porosity of the sand may have affected the volume change, but no information on porosity was presented.

There is some reason to believe that the amount of paste in excess of the voids in the aggregate and the thickness and quality of the paste between particles at their contact points affect shrinkage. I would like to suggest that, if possible, the author translate his parts of sand by weight into parts by volume and calculate the volume of voids in the various sands. Then, with the solid volume of the paste known, I suggest he calculate the excess of paste over the voids and recalculate the thickness of the slurry coatings on the particles on the basis that they came from the excess of paste over the voids.

I question the author's method of calculating the thickness of the slurry. I think it is more reasonable to assume that the thickness of the slurry coatings on the particles is a product of the paste remaining after the voids are filled, granting that there is sufficient paste to completely fill the voids and provide something more than a minimum coating for the particles. The thickness of the slurry coatings would be the excess of the solid volume of the paste over the voids in the aggregate divided by the total surface area of the particles. These thicknesses would be considerably less than those given by the author.

If the author could provide the additional information indicated in my suggestions, I believe it would help greatly to illuminate this question of the influence of sand characteristics on the properties of mortar. I hope he has the necessary basic data and that this research can be carried forward.

It would also help if tests were made and reported on Ottawa sand, graded in the same fashion as the other sands. This would serve as a much better point of reference.

The author has made a most valuable contribution. He has approached the determination of the effects of the characteristics of sand on the properties of mortar from an unusual angle. His results show that the characteristics other than grading exert a powerful influence on the properties of mortar. In 1953 I reported to this Society (**3**) on the effects of variations in the grading of sand on the workability of masonry mortar and their influence on the moistureproofness of masonry walls. Revised requirements for the grading of masonry sand based on sieve fractions were suggested at that time. Masonry sands passing those requirements have produced excellent results in the field, and concrete designers have reported a considerable improvement in the quality of concrete where the concrete sands were selected by applying the same basic theory.

The author's study now makes it entirely clear that the present ASTM tentative specifications for both masonry sand[5] and concrete sand[6] are hopelessly inadequate. More research work needs to be done. The studies of the author and myself have indicated the general direction of that research. The further elimination of inconsistent and ofttimes unfortunate results in both concrete and masonry demand that such research be started as soon as possible.

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[5] Tentative Specifications for Aggregate for Masonry Mortar (C 144 – 52 T), 1958 Book of ASTM Standards, Part 4.

[6] Specifications for Concrete Aggregates (C 33 – 57), 1958 Book of ASTM Standards, Part 4.

MR. DEMPSEY.—The point that Mr. Connor made about the spread in the information and the lack of its correlation seems to be one of the curious characteristics of "mother nature" in her materials. We noticed it ourselves.

In connection with the volumetric measure, I regret that we do not have data to convert to volume along the lines suggested.

TABLE VI.—MORTAR TEST DATA—ROD MILL SAND MORTARS COMPARED TO NATURAL SAND MORTARS AT COMMON SLUMPS.

Sands	Fineness, Modulus	Water-Cement Ratio	Proportion	Per Cent Standard Ottawa	Cracking Pattern
Rod mill	3.0	0.60	1 to 3.15	150	
Rod mill	2.80	0.60	1 to 2.93	169	Many small cracks
Rod mill	2.60	0.60	1 to 2.79	164	
Stream bed	3.0	0.60	1 to 3.34	145	Large and small cracks
Stream bed	2.80	0.60	1 to 3.25	143	
Stream bed	2.60	0.60	1 to 3.03	139	
River	3.0	0.60	1 to 3.20	137	Few small cracks
River	2.80	0.60	1 to 3.00	137	
River	2.60	0.60	1 to 3.13	135	
Beach	3.00	0.60	1 to 3.70	159	
Beach	2.80	0.60	1 to 4.00	147	None
Beach	2.60	0.60	1 to 3.62	142	

In regard to the sands around Lacon and Lockport, we know that they came from similar geologic formations in each case. There were three competing ready-mix organizations from which we got our samples. We knew where their quarries were.

MR. E. L. HOWARD[7] (*by letter*).—Tests we have made of sands have been to measure strength, workability, finishing and cracking pattern of portland-cement mortars. The sands tested were from ten deposits in northern California. Some of the test data are detailed in the accompanying Table VI, including a rod mill sand made from $\frac{1}{2}$ by $\frac{1}{4}$-in. pea gravel. The pea gravel is taken from the same deposit as the sand shown in Table VI as streambed sand.

Our manufactured sand behaved very well. The main fault was with finishing. There is a harshness in rod mill mortars not detected by consistency tests but quickly observed by the finishers. Cement requirements vary with gradings as do strengths. Our studies indicate there is a "just right" grading for each sand. Our rod mill sand was considered best at a 2.80 fineness modulus even though the greater cement economy was at 3.0 to 3.2 fineness modulus.

Perhaps the author's manufactured sand would have fared better at another grading.

The beach sand has a smooth, high polished surface and yields high-strength concrete at good cement economy. Our experience with the smooth surfaced sands quite agrees with the author's.

The tendency to crack does not seem to be related to surface shape or area. But, our tests did not follow up this most important problem. We had hoped the author would shed some light in this direction.

The author has opened the way for

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[7] Chief Testing Engineer, Pacific Cement and Aggregates, Inc., San Francisco, Calif.

more extensive study of sand and the part it plays in concrete mortars.

MR. JOHN G. DEMPSEY (*author's closure*).—As Mr. Connor noted in his discussion, there remains much still to be learned as to the essential qualities of a good fine aggregate.

It is unfortunate that volumetric data on the samples as used is not available. However, the reported method of computing cement-and-water slurry, which is based on the assumption that it fills the voids of a sample in addition to coating sand surfaces gives a relatively good measure of its effective thickness. This is evidenced by a comparative examination of the reported volume changes and slurry thicknesses.

The author does not agree with Mr. Connor that present specifications are "hopelessly inadequate;" rather that what is indicated is that much more investigation of the band of specification tolerance insuring good behavior, as well as further inquiry into the effects of other variables than grading is what is indicated.

A valuable addition to the technique of the situation would undoubtedly be the Goldbeck test for voids suggested by Mr. Gray. The value of a microscope in such a research should also be apparent when the extent of variation of equally graded sands mixed in slurries of constant water-cement ratio is considered.

Was the author's suggestion warranted that the samples of manufactured sands were likely to make more costly mixes? Many generalized tabulations and the opinion of some experienced construction authorities answer in the affirmative. The reason usually given is that the interlocking of the particles of manufactured sand is usually such that more sand is required in a given mix, and also more water per unit of sand to produce a suitable plasticity. Water-cement ratio for a specified strength then demands more cement to go with the additional water; hence greater unit cost for the mixture.

In Mr. Gray's table for Project 49-13 there is an illuminating note that emphasizes the value of air-entrainment to preserve durability. Air was not added in the investigation under discussion, since the particle characteristics themselves were the focal point of the program. The lack of entrained air in some of these samples undoubtedly accounts for their inferior behavior.

In conclusion, the author expresses his thanks to the various discussers and hopes that others will undertake to answer, by further research, some of the uncertainties still remaining.

REFERENCES

(1) A. T. Goldbeck, "Stone Sand for Use as Fine Aggregate," *The Crushed Stone Journal*, June 1951.

(2) A. T. Goldbeck, "A Study of the Effect of Shape of Particle on the Mortar Making Properties of Stone Sand," *The Crushed Stone Journal*, June 1951.

(3) C. C. Connor, "Some Effects of the Grading of Sand on Masonry Mortar," *Proceedings*, Am. Soc. Testing Mats., Vol. 53, p. 933 (1953).

FLEXURAL AND COMPRESSIVE STRENGTH PROPERTIES OF AIR-ENTRAINED CONCRETE WITH AIR-COOLED BLAST-FURNACE SLAG AGGREGATE*

By D. W. Lewis[1] and Fred Hubbard[1]

Synopsis

Previous published reports of investigations of air-cooled slag concrete have been confined to the use of non-airentraining cements. This study was undertaken to provide data on flexural and compressive strengths of air-entrained concretes made with blast-furnace slag. The coarse aggregates were from eight sources having different physical properties. One source having average physical characteristics was selected as the "standard" with which the other slags are compared in six-bag mixes made with a blend of three air-entraining cements. All mixes were repeated on three different mixing days, and strength tests were conducted in accordance with ASTM methods[2] at ages of 7, 14, 28, and 90 days and 1 yr.

Additional variables of aggregate size, cement content, curing condition and use of portland blast-furnace slag cement were studied on mixes with the control or standard aggregate.

The results show no consistent correlation of strength with physical properties of the slag aggregates such as unit weight and abrasion loss. With constant cement content, the variations in aggregate size from ¾ to 2 in. produced essentially the same strengths. The ⅜-in. aggregate concrete had lower strength. Changes in cement content from 4 to 8 sacks per cu yd produced greater effects on the compressive strength than on the flexural strengths. Rate of strength gain after seven days was greater in compression than in flexure, and the compression test is indicated to be the more reproducible.

Moist room and water-immersion curing produced equivalent strengths, but exposure to laboratory air after 24 hr moist curing caused significant strength changes. Although lower in strength at early ages, the portland blast-furnace slag cement was equal to the portland blend after 28 days curing.

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* Presented at the Sixty-first Annual Meeting of the Society, June 22–27, 1958.

[1] Chief Engineer and Director of Research, respectively, National Slag Assn., Washington, D. C.

[2] Method of Test for Flexural Strength of Concrete (Using Simple Beam with Third-Point Loading) (C 78 – 57); Tentative Method of Test for Compressive Strength of Molded Concrete Cylinders (C 39 – 56 T), 1958 Book of ASTM Standards, Part 4.

Comprehensive investigations of the strength properties of air-cooled blast-furnace slag concretes have been reported by Hubbard (**1**)[3] and Hubbard and Williams (**2**). These studies compared the physical properties of the aggregates with compressive and flexural strengths of the concretes made with type I cement. No significant relationships were found between the aggregate and concrete properties, using slags representing the entire range of physical properties to be found with this type of aggregate.

A need existed for additional information, which would include test results with type I-A cements, since air-entrainment is used in the great bulk of present-day concrete. A limited test series was undertaken in the National Slag Assn. Research Laboratory in 1955, using slag aggregates from five sources, a single source of sand, and an air-entraining cement. The 28-day strengths of these concretes are shown in Table I for the three levels of cement content used.

TABLE I.—SUMMARY OF 28-DAY CONCRETE STRENGTHS FROM PRELIMINARY TEST SERIES WITH BLAST FURNACE SLAG AGGREGATES.

Each strength value shown is the average of three tests.

Slag Coarse Aggregate		Compressive Strength, psi			Flexural Strength, psi		
		Cement, sacks per cu yd			Cement, sacks per cu yd		
Unit Weight, lb per cu ft	Los Angeles Abrasion Loss, per cent	5	6	7	5	6	7
72.4	37.7	4550	5630	6160	585	640	680
78.0	26.9	4180	5210	6160	565	665	655
78.6	33.7	4650	5190	6180	655	630	705
84.6	34.9	4160	5220	5760	580	705	730
87.0	34.1	4350	5550	6370	625	690	775
Maximum		4650	5630	6370	655	705	775
Minimum		4160	5190	5760	565	630	655
Average		4380	5360	6110	600	665	710

Despite the range in unit weights of the slag aggregates from 72.4 to 87.0 lb per cu ft, the strength results were quite consistent. The maximum variation from the average for all aggregates was 6 per cent in compression and 9 per cent in flexure. No consistent relationship of strength to unit weight or abrasion loss is indicated.

The test results, however, were based upon single mixes for each slag aggregate and represent only five sources (three specimens, all from the same batch for each test). A more comprehensive test series, to include additional aggregate sources representing a wider range of weight and abrasion and with mixes run in triplicate was felt to be desirable. Accordingly, the test series reported in this paper were undertaken.

SCOPE

The major portion of the study consisted of a comparison of the flexural and compressive strength properties of concrete made with type I-A cement and with slag aggregate from eight commercial sources (mix Nos. 1–8). These slags covered the entire range of unit weights and abrasion losses ordinarily encountered. Each mix was repeated on three different days, with strength tests conducted at ages of 7, 14, 28, 90 days and 1 yr. Three compression and five flexural tests were run at each age to represent the three replications of a given mix. A cement content of 6 sacks per cu yd, $1\frac{1}{2}$-in. maximum size aggregate, and a 2-in. slump were used throughout these mixes.

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[3] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 1156.

TABLE II.—CHEMICAL AND PHYSICAL PROPERTIES OF CEMENTS.

Source	Type I-A			Type IS-A
	A	B	C	D
Chemical analyses				
SiO_2	21.09	20.89	20.72	25.0
Fe_2O_3	2.08	3.55	4.12	2.8
Al_2O_3	5.09	6.33	6.04	9.6
CaO	64.03	64.25	64.20	55.6
MgO	1.08	1.24	...	4.3
SO_3	2.00	2.12	1.99	1.9
Ignition loss	0.08	0.81	0.50	0.7
Autoclave expansion, per cent	0.061	0.060	0.035	0.030
Specific surface area, sq cm per g	1725	1729	1900	2340
Air (in mortar), per cent	17.1	17.9	18.6	19.2
Initial setting time, hr-min	1-30	1-28	1-25	1-40
Final setting time, hr-min	5-18	5-13	5-3	...
Compressive strength (mortar cubes), psi				
1 day	800	700	1150	1110
3 day	1766	1866	1950	2200
7 day	2733	2150	2700	3620
28 day	...	...	...	5380
Passing No. 325 sieve, per cent	...	84.2	...	97.0

One of the slag coarse aggregates (No. 5), having approximately average physical characteristics, was selected as a "standard" material, and a mix was made with it on each mixing day. This "standard" mix was used as the basis for comparison of results in many cases, particularly for the investigations of other variables outlined below.

In addition to the major variable of source of slag aggregate, limited test series were conducted (all with slag No. 5) to determine the effect of the following:

Cement Content and Cement Type.—The type I-A cement was used in compressive and flexural strength and durability tests (freezing-and-thawing) on 4-, 5-, 6-, 7-, and 8-sack mixes (mix Nos. 5, 9 to 12). Strength tests were run on a 6-sack mix made with type IS-A cement (mix No. 13).

Aggregate Size.—Slag No. 5 was used with maximum sizes of 2-, $1\frac{1}{2}$-, $\frac{3}{4}$- and $\frac{3}{8}$-in. in 6-sack mixes for both flexural and compressive strength tests (mix Nos. 5, 14 to 16).

Type of Curing.—The standard 6-sack mix with slag No. 5 was tested for strength properties with three types of curing—standard moist room, immersion in lime water, and exposure to laboratory air after removal from the molds (mix Nos. 5, 17, 18).

MATERIALS

The materials used throughout the investigation were representative of currently produced commercial materials as described below.

Cement:

Type I-A cement was obtained by blending, in equal proportions, three locally available cements of this type. The type IS-A cement was from a single source. Chemical and physical characteristics of these cements are shown in Table II.

Coarse Aggregate—Blast-Furnace Slag:

The eight slag aggregates tested were commercially produced aggregates, selected to provide a wide range in unit weight and abrasion loss. Physical characteristics of these materials are given in Table III. The slags ranged in unit weight (dry rodded basis) from 71.2 to 88.0 lb per cu ft, and from 29.4 to 48.9 per cent in Los Angeles abrasion loss. The abrasion loss of 48.9 per cent is unusually high for slag. This material, from a small, one-furnace operation, was included to determine the effect of abnormally high abrasion loss on concrete strengths.

TABLE III.—PROPERTIES OF AGGREGATES.

PHYSICAL PROPERTIES

Property	Blast-Furnace Slag Coarse Aggregates								Natural Sand
	1	2	3	4	5	6	7	8	
Weight, lb per cu ft (dry-rodded basis)[a]	71.2	73.8	74.2	81.0	82.8	86.6	87.6	88.0	111.0
24 hr absorption, per cent	3.9	3.3	3.0	3.0	2.5	1.2	2.1	1.8	2.0
Dry bulk specific gravity	2.21	2.13	2.25	2.26	2.29	2.39	2.42	2.40	2.57
Voids, compact, per cent	48.3	44.5	47.2	42.5	42.1	42.0	42.0	41.2	30.8
Los Angeles abrasion loss, per cent (A grading)	29.4	36.6	35.0	48.9	33.0	43.0	33.8	30.4	...
Na_2SO_4 soundness loss, per cent (5 cycles)	...	...	...	...	...	...	...	...	6.6

GRADINGS

Sieve Size	Total Per Cent Passing				
	Blast-Furnace Slag Coarse Aggregate				Sand
2-in.	...	[b]100	...	...	...
1½-in.	100	72	...	...	...
1-in.	62	45	...	...	...
¾-in.	43	31	[b]100	...	...
½-in.	24	18	55	[b]100	...
⅜-in.	14	10	33	92	100
No. 4	0	0	0	15	98.6
No. 8	...	...	...	0	83.8
No. 16	...	...	...	...	65.8
No. 30	...	...	...	...	51.0
No. 50	...	...	...	...	26.0
No. 100	...	...	...	...	5.8
No. 200	...	...	...	...	1.2
Fineness modulus	7.43	7.87	6.67	6.08	2.69

[a] Unit weights based on 1½-in. maximum size grading shown.

[b] These gradings used only with slag No. 5 in test series to determine effect of maximum size of coarse aggregate.

NOTE.—For designation of ASTM tests used see footnote 4.

Fine Aggregate—Natural Sand:

A locally available concrete sand was used throughout all mixes to avoid any variations from the fine aggregate characteristics. The sand used was of good quality, as shown by the physical characteristics in Table III.

Test Procedures

All of the materials were obtained in sufficient quantities to insure an adequate supply for the entire study. Standard ASTM procedures were followed in tests on the materials.[4] Concrete mixes were proportioned to provide a constant dry-rodded volume of coarse aggregate per unit volume of concrete, b/b_0, of 0.76 for 2-in. aggregate, 0.73 for $1\frac{1}{2}$-in., 0.62 for $\frac{3}{4}$-in., and 0.43 for $\frac{3}{8}$-in. material. The water-cement ratio was permitted to vary as necessary to provide a constant slump of approximately 2 in.

Mixing:

The aggregates were used in the existing moisture condition, with quantities adjusted for the actual moisture contents. Mixing was done in a Lancaster tub-type mixer, with 4.5 cu ft of concrete per batch. The aggregates were placed in the mixer with part of the mixing water (approximately one half) and the mixer revolved several turns. It was then stopped for 5 min to allow absorption by the aggregates to take place.

The cement was then added and the remainder of the water added while mixing was continued. After approximately 4 min of additional mixing, the slump, yield, Kelly-ball penetration, and air content determinations were made using standard procedures. Air content was determined by the volume method, ASTM Tentative Method C 173 – 55 T.[5]

Molding and Curing:

All specimens were molded in accordance with standard ASTM procedures, with five 6 by 12-in. cylinders[6] and three 6 by 6 by 40-in. beams made from each batch. Three such batches were used for each mix composition tested. For freezing-and-thawing tests two 3 by 4 by 16-in. beams were molded from the batches that were to be included in this part of the study.

Specimens were covered with wet burlap for 24 hr, followed by removal from the molds. Moist-cured specimens were placed in a moist room at 72 ± 1 F and approximately 100 per cent relative humidity until tested. Specimens to be water cured were immersed in a tank of lime water in the moist room. Dry curing was accomplished by exposure to laboratory air at approximately 72 F, but without humidity control, following removal from the molds.

Strength Tests:

Compression tests were run in a Tinius-Olsen hydraulic machine and flexure tests in a Rainhart hydraulic beam breaker using third-point loading on an 18-in. span. ASTM procedures were followed.[2]

Freezing-and-Thawing Tests:

Specimens for freezing-and-thawing tests were moist cured until 26 days old and then immersed in water for 48 hr prior to the first cycle of freezing. A Conrad automatic freeze-thaw chamber

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[4] Tentative Method of Test for Unit Weight of Aggregate (C 29 – 55 T); Method of Test for Specific Gravity and Absorption of Coarse Aggregate (C 127 – 42); Methods of Test for Specific Gravity and Absorption of Fine Aggregate (C 128 – 57); Method of Test for Voids in Aggregate for Concrete (C 30 – 37); Method of Test for Abrasion of Coarse Aggregate by Use of the Los Angeles Machine (C 131 – 55); Tentative Method of Test for Soundness of Aggregates by Use of Sodium Sulfate or Magnesium Sulfate (C 88 – 56 T); Method of Test for Sieve Analysis of Fine and Coarse Aggregates (C 136 – 46); 1958 Book of ASTM Standards, Part 4.

[5] Tentative Method of Test for Air Content of Freshly Mixed Concrete by the Volumetric Method (C 173 – 55 T), 1958 Book of ASTM Standards, Part 4.

[6] Method of Making and Curing Concrete Compression and Flexure Test Specimens in the Laboratory (C 192 – 57), 1958 Book of ASTM Standards, Part 4.

TABLE IV.—MIX PROPORTIONS AND PROPERTIES OF FRESH AND HARDENED CONCRETE.

Mix	Coarse Aggregate		Quantities per cu yd, Dry			Net Water, gal per sack	Weight, lb per cu ft	Consistency		Air Content, per cent	Hardened	
	Slag Number	Maximum Size, in.	Cement, sacks	Coarse Aggregate, lb	Fine Aggregate, lb			Slump, in.	Kelly Ball, in.		Oven Dry Weight, lb per cu ft	24 hr Absorption, lb per cu ft
No. 1	1	1½	5.95	1393	1346	5.8	135.0	2.1	1.2	4.9	125.5	7.3
No. 2	2	1½	5.95	1443	1222	5.2	131.2	2.0	1.0	4.6	125.6	6.0
No. 3	3	1½	5.97	1454	1295	5.8	135.3	2.2	1.2	4.3	126.9	6.9
No. 4	4	1½	6.05	1610	1193	5.7	137.6	2.0	1.3	4.5	129.6	6.6
No. 5	5	1½	6.00	1631	1159	5.3	135.9	2.0	1.1	4.1	129.7	6.1
No. 6	6	1½	5.92	1684	1138	5.1	136.1	2.1	1.1	4.7	129.8	6.4
No. 7	7	1½	6.02	1731	1157	5.2	139.3	2.0	1.1	4.5	132.9	6.3
No. 8	8	1½	6.03	1746	1140	5.0	138.9	2.0	1.2	4.4	133.8	5.8
No. 9	5	1½	4.05	1651	1304	7.4	134.9	1.9	0.9	4.9	127.2	6.9
No. 10	5	1½	5.01	1636	1235	6.4	135.6	2.0	1.0	4.3	128.5	6.7
No. 11	5	1½	6.93	1616	1089	4.8	136.5	2.0	1.1	4.0	129.3	6.3
No. 12	5	1½	7.81	1595	1014	4.5	136.4	2.1	1.2	3.8	128.9	6.4
No. 13[a]	5	1½	5.91	1609	1134	5.4	134.0	2.0	0.9	5.1	128.0	6.3
No. 14	5	2	5.98	1669	1131	5.2	136.1	2.2	0.9	4.1	128.3	6.4
No. 15	5	¾	6.10	1341	1452	5.8	137.6	2.1	1.6	4.4	129.5	6.8
No. 16	5	⅜	6.10	929	1738	6.4	134.2	2.7	2.2	6.1	124.6	8.1
No. 17[b]	5	1½	5.99	1629	1154	5.2	135.2	2.0	1.0	4.4	128.6	6.3
No. 18[c]	5	1½	5.99	1629	1154	5.1	135.4	2.1	0.9	4.5	129.5	6.8

[a] Portland blast-furnace slag-cement used.

[b] Curing by immersion in lime water.

[c] Dry curing exposure to laboratory air.

Values given are the averages for three repetitions of each mix except in the case of mix No. 5 which was repeated 18 times.

TABLE V.—EFFECT OF SLAG COARSE AGGREGATE ON STRENGTH OF CONCRETE.

(Type I-A cement in 6-sack mixes with 1½-in. aggregate, moist cured. Strengths shown are averages for three compression and five flexure tests except for mix No. 5 where 18 compression and 30 flexure tests were conducted at each age.)

Mix	Coarse Aggregate			Compressive Strength, psi					Flexural Strength, psi				
	Aggregate Number	Unit Weight, lb per cu ft	Abrasion Loss, per cent	7 days	14 days	28 days	90 days	1 yr	7 days	14 days	28 days	90 days	1 yr
No. 1	1	71.2	29.4	3950	4530	5110	5740	6170	535	620	645	675	790
No. 2	2	73.8	36.6	4350	5350	6000	6730	7100	575	660	665	690	725
No. 3	3	74.2	35.0	4040	4920	5610	5860	6380	560	590	645	665	715
No. 4	4	81.0	48.9	4390	5240	6010	6380	7170	565	635	635	605	710
No. 5	5	82.8	33.0	4320	5120	5830	6580	6980	540	615	640	665	710
No. 6	6	86.6	43.0	3750	4700	5620	6140	6500	495	580	605	665	690
No. 7	7	87.6	33.8	4120	5240	5760	6760	7170	560	635	680	735	735
No. 8	8	88.0	30.4	4190	5050	5880	6630	7030	590	670	660	725	750
Maximum strength				4390	5350	6010	6760	7170	590	670	680	735	790
Minimum strength				3750	4530	5110	5740	6170	495	580	605	605	690
Average strength, psi				4140	5020	5730	6350	6810	550	625	645	680	730

was used for the tests, operating on a 2-hr cycle. Freezing in air to 0 F and thawing in water to 40 F were each accomplished in about 1 hr. These procedures follow the requirements of ASTM Tentative Method C 291 – 57 T.[7] Effect of the freezing and thawing was determined by the dynamic modulus of elasticity test, ASTM Tentative Method C 215 – 55 T.[8]

DISCUSSION OF DATA

Data on the mix proportions used in the study and the characteristics of both the fresh and hardened concretes are shown in Table IV. Average values for three repetitions of the mixes are given, except in the case of the standard mix (No. 5) which was repeated on 18 different mixing days. The individual mix repetitions were very close to the average values reported in all cases. Cement contents varied but slightly, deviating from the design value by less than 0.1 sack per cu yd in all mixes except No. 12. In this case the actual cement content was 7.8 sacks instead of the design figure of 8.0 sacks.

Consistency of the concrete mixes was quite uniform, with Kelly-ball penetration averaging 53 per cent of the slump measured in inches for the $1\frac{1}{2}$-in. aggregate. This varied with aggregate size, being 41 per cent for 2-in. aggregate, 76 per cent for $\frac{3}{4}$-in. and 82 per cent with $\frac{3}{8}$-in. aggregate. These data indicate a straight-line relationship between the ratio of Kelly-ball penetration to slump and the maximum size of aggregate in inches.

Relationship of Concrete Strength to Physical Properties of Slag Aggregate:

Compressive and flexural strengths of the concretes made with each of the slag coarse aggregates are given in Table V. All of the mixes represented here were made with $1\frac{1}{2}$-in. maximum size aggregate and the blend of type I-A cements and were cured in the moist room until time of test.

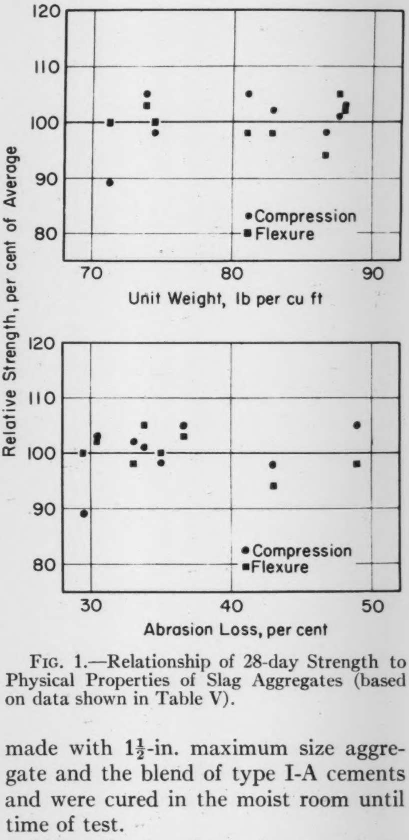

FIG. 1.—Relationship of 28-day Strength to Physical Properties of Slag Aggregates (based on data shown in Table V).

Although the slag aggregates vary from 71.2 to 88.0 lb per cu ft in unit weight and from 29.4 to 48.9 per cent Los Angeles abrasion loss, concrete strengths were quite consistent. In only two cases did the strength of any mix differ more than 10 per cent from the average of all mixes. These were the 28-day compressive strength of mix 1 and the 90-day flexural strength of mix 4, both of which are 11 per cent below the average for all mixes.

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[7] Tentative Method of Test for Resistance of Concrete Specimens to Rapid Freezing in Air and Thawing in Water (C 291 – 57 T), 1958 Book of ASTM Standards, Part 4.

[8] Tentative Method of Test for Fundamental Transverse, Longitudinal, and Torsional Frequencies of Concrete Specimens (C 215 – 55 T), 1958 Book of ASTM Standards, Part 4.

TABLE VI.—EFFECT OF CEMENT CONTENT AND CEMENT TYPE ON STRENGTH OF SLAG CONCRETE.

(Slag No. 5 used in 1½-in. maximum size, all mixes moist cured. Strengths shown are averages for three compression and five flexure tests except mix No. 5 where 18 compression and 30 flexure tests were conducted at each age.)

Mix	Cement		Compressive Strength, psi					Flexural Strength, psi				
	Type	Sacks per cu yd	7 days	14 days	28 days	90 days	1 yr	7 days	14 days	28 days	90 days	1 yr
No. 9......	I-A	4.0	2280	3080	3780	4070	4750	420	480	560	540	615
No. 10......	I-A	5.0	3270	4160	5120	5310	5930	545	575	560	625	635
No. 5......	I-A	6.0	4320	5120	5830	6580	6980	540	615	640	665	710
No. 11......	I-A	6.9	4800	5550	6540	6990	7470	635	655	690	755	785
No. 12......	I-A	7.8	5540	6510	6960	7450	7920	595	740	715	785	805
No. 13......	IS-A	5.9	3650	4820	5770	6600	7040	525	575	625	720	770

TABLE VII.—VARIATION IN RELATIVE STRENGTHS WITH DIFFERENT CEMENTS AND CEMENT CONTENTS.

All mixes made with slag No. 5, 1½-in. maximum size. Values calculated from data in Table VI.

Cement		Relative Strength, per cent of standard mix									
Type	Sacks per cu yd	Compression					Flexure				
		7 days	14 days	28 days	90 days	1 yr	7 days	14 days	28 days	90 days	1 yr
I-A............	4.0	53	60	65	62	68	78	78	88	81	86
I-A............	5.0	76	81	88	81	85	101	88	88	94	89
I-A............	6.0	100	100	100	100	100	100	100	100	100	100
I-A............	6.9	111	108	112	106	107	117	106	108	114	110
I-A............	7.8	128	127	120	113	113	110	120	112	118	113
IS-A...........	5.9	84	94	99	100	101	97	94	98	108	108

To illustrate the relationship of strength to the physical properties of the aggregate, the 28-day relative strengths (as percentages of the average for all mixes) are plotted against unit weight and abrasion loss of the aggregates in Fig. 1. The data show no consistent variation of strength with either of these physical properties. The lightest slag had the lowest compressive strength but had average flexural strength. The next lightest slag had higher than average strengths in both compression and flexure, while the highest compressive strength of all was obtained with an average weight slag having an abrasion loss of nearly 49 per cent.

The results of these tests confirm the findings of previous research work (**1**, **2**), showing that neither unit weight nor abrasion resistance of blast-furnace slags can be used to indicate strengths of concrete made with them. With average 28-day strengths of 5730 and 645 psi in compression and flexure, respectively, it seems doubtful that there is any practical significance to any strength differences obtained in this phase of the study.

Effect of Cement Content and Cement Type:

The results of tests with varying contents of type I-A cement and with the IS-A cement are shown in Table VI. Slag-aggregate No. 5 in the $1\frac{1}{2}$-in. maximum size was used in these mixes, which were cured in the moist room. To facilitate comparison of these mixes, relative strength values were computed, using the standard 6-sack mix (mix No. 5) as the basis for comparison (100 per cent value). These values are tabulated in Table VII.

TABLE VIII.—EFFECT OF AGGREGATE SIZE AND CURING METHOD ON STRENGTH OF SLAG CONCRETE.

(6-sack mixes made with slag No. 5, type I-A cement. Strengths shown are averages for three compression and five flexure tests except for mix No. 5 where 18 compression and 30 flexure tests were conducted.)

Mix	Maximum Size of Aggregate, in.	Curing	Compressive strength, psi					Flexural strength, psi				
			7 days	14 days	28 days	90 days	1 yr	7 days	14 days	28 days	90 days	1 yr
No. 14......	2	Moist	4350	5270	5620	6180	6490	570	625	615	715	735
No. 5.......	$1\frac{1}{2}$	Moist	4320	5120	5830	6580	6980	540	615	640	665	710
No. 15......	$\frac{3}{4}$	Moist	4120	5060	5620	6260	7120	550	610	650	725	745
No. 16......	$\frac{3}{8}$	Moist	2750	3600	4320	4920	5430	505	560	565	660	620
No. 17......	$1\frac{1}{2}$	Water	4240	5230	5890	6300	7100	575	635	655	705	725
No. 18......	$1\frac{1}{2}$	Air	3920	4620	5090	5210	5050	440	445	420	635	675

Variation in the cement content from 4.0 to 7.8 sacks per cu yd produced compressive strengths at 7 days ranging from 53 to 128 per cent of 6-sack mix strengths. This range decreased with age, however, being only 65 to 120 per cent at 28 days and 68 to 113 per cent after 1 yr of curing. The change in compressive strength from variation in cement content is therefore dependent upon the age at the time of test, with the least effect shown at later ages.

The relative flexural strength values are somewhat erratic but exhibit the same general trend as found for the compressive strength. However, the most apparent factor is the smaller range of values in flexure than in compression, the maximum range in flexure being from 78 to 120 per cent of the strength of the standard 6-sack mix. The flexural strength is obviously less sensitive to changes in the cement content than is compressive strength, and this is particularly evident for decreases below the 6-sack per cu yd level.

The mix made with type IS-A cement (No. 13) showed lower compressive strengths at 7 and 14 days than did the comparable mix with type I-A cement (No. 5). At all later ages the strengths were practically identical. In flexure, slightly higher strengths were obtained with the type IS-A cement at 90 days and 1 yr.

Effect of Aggregate Size and Method of Curing:

Test results for the series to determine the effects of variations in size of the coarse aggregate and the effect of the curing method are given in Table VIII. Figure 2 shows, in graphical form, the relative strengths for the various maximum sizes of slag aggregate, using the standard mix (No. 5) with $1\frac{1}{2}$-in. slag as the basis for the comparison. For both compression and flexure, strengths obtained with either 2- or $\frac{3}{4}$-in. aggregate (mix Nos. 14 and 15) were very close

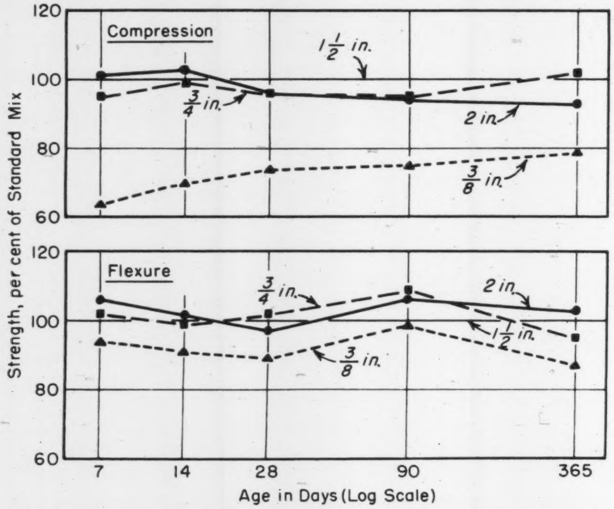

FIG. 2.—Effect of Maximum Size of Slag Aggregate on Strength (based on data in Table VIII, with mix No. 5 made with 1½ in. aggregate used as 100 per cent for comparison).

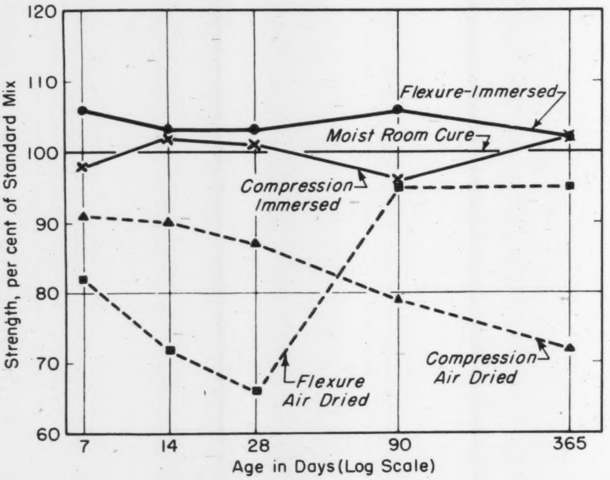

FIG. 3.—Effect of Curing Condition on Relative Strength of Six-Sack Mixes (based on data in Table VIII with moist-room curing, mix No. 5, taken as the 100 per cent value for comparisons).

to the 100 per cent line representing the 1½-in. size. No significant differences are indicated for these three sizes. At constant cement content (6 sacks per cu yd), however, use of ⅜-in. aggregate (mix No. 16) reduced the compressive strengths to 64 per cent of the standard value at 7 days and 78 per cent at 1 yr. A gradual increase of relative strength is apparent with increased curing time.

TABLE IX.—RESULTS OF FREEZING-AND-THAWING TESTS.

Concrete contained slag No. 5, 1½-in. maximum size and type I-A cement.

Mix	Cement Content, sacks per cu yd	Air Content, per cent	Water Cement Ratio, gal per sack	Dynamic Modulus, E, after 303 cycles of Freezing and Thawing,[a] per cent of original value
No. 9....	4.0	4.9	7.4	101
No. 10....	5.0	4.3	6.4	101
No. 5....	6.0	4.1	5.3	97
No. 11....	6.9	4.0	4.8	100
No. 12....	7.8	3.8	4.5	99

[a] Freezing in air at 0 F; thawing at 40 F in water. Each mix represented by two 3 by 4 by 16-in. beams.

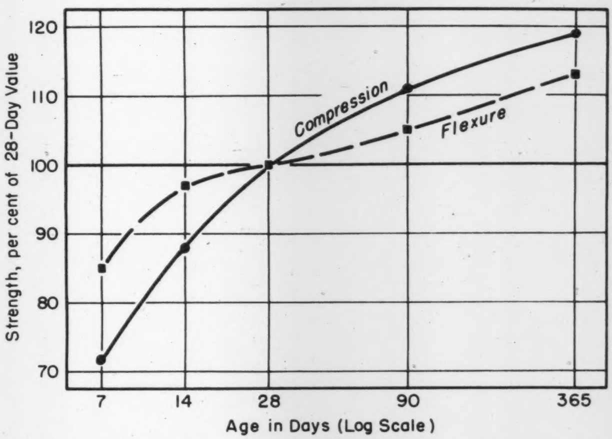

FIG. 4.—Rate of Strength Gain, Average for Six-Sack Mixes With Eight Sources of Slag Aggregates (based on data in Table V).

Reduction in flexural strength was much less pronounced, with the relative values for the ⅜-in. material ranging from 87 to 99 per cent at various ages.

Differences in curing method plotted in Fig. 3 likewise show differing effects on compressive and flexural strengths. Moist-room curing (the basis of comparison or 100 per cent level in the graph) and curing by immersion in water are shown to be essentially equal. Drying in laboratory air after removal from the molds produced a steadily decreasing relative compressive strength value, ranging from 91 per cent at 7 days to 72 per cent after 1 yr. Relative flexural strength decreased with drying time to a minimum value of 66 per cent at 28 days. The 90 day and 1-yr values, however, were 95 per cent of the standard. This indicates little effect of curing condition on flexural strength at later ages.

Durability Tests:

Freezing-and-thawing test results on concretes made with 1½-in. maximum size slag aggregate and varying contents of type I-A cement are shown in Table IX. These tests show no significant change in the dynamic modulus of elasticity for any of the mixes, varying from 4.0 to 7.8 sacks of cement per cu yd, when subjected to 303 cycles of freezing in air and thawing in water.

Rate of Strength Gain and Reproducibility of Test Results:

Throughout the data presented, it is evident that the compressive strengths showed a rather uniform increase with age, while the flexural strengths tended to be somewhat erratic, particularly in the range of 14 to 90 days age. In several cases the flexural strengths obtained for a mix (average for 5 beams representing three repetitions of the mix made in one week) indicate no gain in strength either between 14 and 28 days or between 28 and 90 days. Data contained in many published papers (3–6) show that such trends in flexural strength are frequently encountered in tests with a wide variety of cements and aggregates.

TABLE X.—STRENGTH DATA FOR MIX NO. 5 BY INDIVIDUAL WEEKS OF MIXING.
Strength values shown for each age are the averages for three cylinders or five beams representing three repetitions of the mix made in one week.

Mix	Compressive Strength, psi					Flexural Strength, psi				
	7 days	14 days	28 days	90 days	1 yr	7 days	14 days	28 days	90 days	1 yr
5 A, B, C	4350	4980	5810	6960	6960	525	620	635	670	740
5 D, E, F	4220	4830	5920	6580	6980	525	600	620	690	700
5 G, H, J	4260	5110	5740	6400	6690	565	620	685	625	695
5 K, L, M	4340	5220	5850	6420	7220	550	630	615	680	680
5 N, P, R	4300	5220	5710	6400	7040	525	610	620	655	705
5 S, T, U	4470	5410	5940	6740	7020	560	600	665	670	750
Maximum weekly average	4470	5410	5940	6960	7220	565	630	685	690	750
Minimum weekly average	4220	4830	5710	6400	6690	525	600	615	625	680
Average $\overline{X}$	4320	5120	5830	6580	6980	540	615	640	665	710
Total number of tests	18	18	18	18	18	30	30	30	30	30
Standard deviation, σ, psi	209	265	195	337	305	31	30	51	53	41
Coefficient of variation, per cent	5.3	5.2	3.3	5.1	4.4	5.7	4.9	8.0	7.9	5.8

The average rates of strength gain for mix Nos. 1 to 8, representing all eight slag aggregates used, are shown in Fig. 4, where strength as a percentage of the 28-day value is plotted against the age at time of test. During the entire period from 7 days to 1 yr, compressive strength varied from 72 to 119 per cent of the 28-day value. The strength range in flexure for the same period was 85 to 113 per cent—a total variation of only 28 per cent as compared to 47 per cent for compressive strength. In addition, the curve for compression is uniform, while the rate of gain in flexure is particularly low between 14 and 90 days. During the 14 to 90-day period the compressive strength increased by 23 per cent while the increase in flexural strength was only 8 per cent—approximately one third as great.

Similar differences between flexure and compression in the rates of strength gain with age have been shown in a number of previous investigations, including the early work of Abrams (7). Jackson (8) reports results of tests on eight cements (representing both types I and II) with and without air-entrainment. His data indicate wide differences in rate of strength gain, apparently dependent upon type and chemical composition of the cement. Between 7 days and 1 yr, strength gains in compression varied from about four to less than two times the gain in flexure for the different cements (basing calculations of strength gains on the percentages of the 28-day strength).

The percentage values shown in Fig. 4 for all of the slag aggregates are practically identical (within 2 per cent in all cases) to those for the average obtained for all repetitions of the standard mix (No. 5), detailed data for which are shown in Table X. These mixes also indicate, in some cases, erratic weekly average flexural strength values, in the same manner that was found in the other mixes. The results are indicated to be the same, on the average, whether a number of test rounds with one aggregate or one test round with each of several aggregates was used. The percentage change in strength between two test ages was consistently less in flexure than in compression. Both this low rate of strength gain in flexure and the erratic flexural strengths in individual tests appear to be due to characteristics of the cement and of the flexural test itself.

The variability of the tests as measured by the standard deviation and coefficient of variability shown in Table X are also of interest. These values show that the flexural strength tests were characterized by greater variability than was compression. For a given number of individual observations, confidence limits for flexure would have a greater spread, as a percentage of the measured value, than for compression.

Even if a coefficient of variation of 5 per cent could be maintained in the flexural strength test, confidence limits at the 90 per cent level for 28-day strength would be so wide that the average strengths at both 14 and 90 days would fall within them for less than six or seven tests. The confidence limits themselves would overlap unless some 16 or more tests were conducted. Certainly no significance can be attributed to the variations encountered in flexural strength results for the individual mixes in this study between 14 and 90 days of age.

The results obtained confirm the data of previous investigations regarding two important points in flexural strength tests of concrete:

(*a*) It should not be assumed that the rate of strength gain in flexure is comparable to that in compression for any concrete, irrespective of materials used,—they may be vastly different; and

(*b*) Unless a large number of test specimens is used, the variability inherent in the test may lead to erroneous conclusions regarding the increase in strength with age of the specimens.

It should also be noted that the different rates of strength gain result in varying ratios of compressive strength to flexural strength. In this study the compressive strength of the standard mix (Table X) was 8.0 times the flexural strength at 7 days while at 90 days this ratio was 9.9. Therefore, if estimates of the flexural strength are to be made from compression tests, the proper ratio should be established for the particular test age being considered.

CONCLUSIONS

Based upon the tests conducted and the results reported and discussed above, the following conclusions are drawn:

1. No consistent relationship was found between either the abrasion resistance or unit weight of blast-furnace slag aggregates and strength properties of concrete in which they were used.

2. Changes in cement content produced greater changes in both compressive and flexural strength at early ages. Differences decreased with additional curing.

3. Flexural strength was less affected by changes in cement content than was the compressive strength.

4. The type IS-A cement produced lower strengths at 7 and 14 days than did the type I-A cement; strengths were essentially the same at 28 days and later ages.

5. Variations in maximum size of the slag coarse aggregate from $\frac{3}{4}$ to 2 in. in 6-sack mixes had no marked effect on

the strength in either compression or flexure.

6. Use of $\frac{3}{8}$-in. top size aggregate with the same cement factor, 6 sacks per cu yd, decreased the compressive strength 22 to 36 per cent at different ages but caused little apparent change in the flexural strength.

7. Moist room curing and immersion in water were equally effective curing methods, producing essentially the same strengths in both compression and flexure.

8. Exposure of specimens to laboratory air after removal from the molds caused a marked decrease in compressive strength, with relative strength decreasing continuously with length of exposure. Flexural strengths dropped sharply through 28 days age; but tests at 90 days and 1 yr showed little effect from the lack of proper curing.

9. Air-entrained concrete made with an average slag and cement contents varying from 4 to 8 sacks per cu yd showed excellent durability in 303 cycles of freezing in air and thawing in water.

10. The rate of increase in flexural strength with increased curing time was much smaller than that for compressive strength.

11. The flexural strength test was inherently more variable than the compression test, and

12. The data confirm the results of previous investigations and indicate that conclusions regarding increases in flexural strength with age, based on the small number of tests usually conducted and for short periods of additional curing, may be misleading.

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DISCUSSION

MESSRS. STANTON WALKER, DELMAR L. BLOEM, AND RICHARD D. GAYNOR (*by letter*).[1]—We are particularly interested in the authors' findings on the effects of maximum size of aggregate and moisture condition of test specimens on the compressive and flexural strength of concrete made with air-cooled blast-furnace slag as coarse aggregate. Similar results have been found with gravel and crushed stone in the Joint Research Laboratory of the National Sand and Gravel Assn. and the National Ready Mixed Concrete Assn. at the University of Maryland. Our tests on the effect of moisture condition confirm the authors' findings. The data are published elsewhere and will not be discussed here. The reader is referred to "Effects of Curing and Moisture Distribution on Measured Strength of Concrete," by Walker and Bloem.[2]

That larger aggregates produce higher concrete strengths than smaller ones, with other conditions comparable, has been an axiom ever since there has been a developed concrete technology. This "natural law" appears to have been universally accepted without reservation, particularly in the case of compressive strength. The authors' tests, our own, and those of others suggest that this relationship should be reexamined. The data not only fail always to show improved strength for larger aggregate sizes but in some cases actually indicate reductions. This unorthodox relationship came to our attention as a by-product of a number of different investigations, as a result of which we decided to examine the question in detail.

Studies of $\frac{3}{8}$-in. to $2\frac{1}{2}$-in. Maximum Sizes (Series 155-B):

Concrete of one cement factor was made with two aggregates in four different maximum sizes. Both 6 by 12-in. cylinders and 6 by 6 by 36-in. beams were molded. The concrete was designed to contain 6 sacks of cement per cu yd and to have a slump of 2 to 3 in. It contained no purposely entrained air. A single ratio of fine to coarse aggregate was used for each size of each aggregate, selected by conventional criteria to produce workable but not over-sanded mixtures. Compressive and flexural strengths were measured at 28 days.

The results are given in the accompanying Table XI and Fig. 5. Note that neither compressive nor flexural strength showed any significant increase beyond a maximum size of $\frac{3}{4}$ in. and that, in compression, the $2\frac{1}{2}$-in. aggregate gave substantially lower strengths than any of the three smaller sizes. This was in spite of normal reductions in mixing water with increased size, as shown in the table and the lower portion of the figure.

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[1] Director of Engineering, Associate Director of Engineering and Associate Research Engineer, respectively, National Sand and Gravel Assn., and National Ready Mixed Concrete Assn., Washington, D. C.

[2] *Proceedings*, Highway Research Board, Vol. 36 (1957). (Reprinted as a joint publication, Nat. Sand and Gravel Assn. and Nat. Ready Mixed Concrete Assn.)

Tests with Larger Specimens (Series 163-A):

The relationship found was unexpected and we sought for an explanation. The thought suggested itself that the 6-in. cylinders and the 6 by 6-in. beams may not have been large enough in cross-section to accommodate the larger sizes of aggregate without detriment to measured strength. There was also the possibility that the single ratios of fine to coarse aggregate were not optimum in spite of their having been selected by conventional methods. To check these possibilities, tests were made using 8-in. diameter cylinders and 8 by 8-in. beams as well as the standard 6-in. sizes. Three different ratios of fine to coarse aggregate were used for each of the same four maximum sizes—$\frac{3}{8}$, $\frac{3}{4}$, $1\frac{1}{2}$ and $2\frac{1}{2}$ in.—used in the earlier study. Only one coarse aggregate was tested. The cement factor was 6 sacks per cu yd and the slump 2 to 3 in. The data are given in Table XII and the accompanying Figs. 6 and 7, the figures showing only the compression tests.

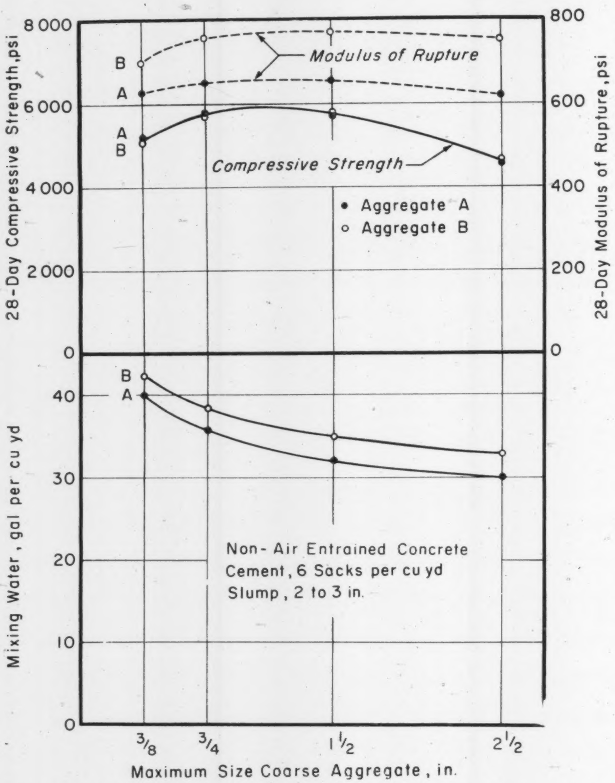

FIG. 5.—Effect of Size of Coarse Aggregate on Strength and Mixing Water Requirement of Concrete Group 2 (Series 155-B).

TABLE XI.—CHARACTERISTICS OF FRESH CONCRETE AND RESULTS OF STRENGTH TESTS (SERIES 155-B).

Each value average for 3 batches of concrete mixed on different days; compressive strengths averages for three 6 by 12 in. cylinders; moduli of rupture averages for 6 tests of 6 by 6 in. beams (2 breaks from 36-in. long beam from each batch).

Maximum Size Aggregate, in.	Sand Total Absolute Volume Aggregate, per cent	Fineness Modulus, Total Aggregate	b/b_o	Cement, sacks per cu yd	Water		Slump, in.	Air, per cent[a]	28-day Strengths, psi	
					gal per cu yd	gal per sack			Compression	Flexure
COARSE AGGREGATE A										
3/8..............	47.0	4.42	0.58	5.93	39.9	6.74	2.7	2.3	5215	629
3/4..............	37.4	5.14	0.68	5.96	35.7	5.98	2.7	1.5	5790	651
1½..............	30.2	5.85	0.77	6.00	32.1	5.35	2.5	0.8	5705	656
2½..............	23.3	6.57	0.82	6.01	29.9	4.98	2.1	0.1	4560	620
COARSE AGGREGATE B										
3/8..............	53.2	4.22	0.58	5.92	42.2	7.13	2.4	3.0	5045	698
3/4..............	43.5	4.89	0.68	5.93	38.3	6.46	2.5	1.8	5660	756
1½..............	33.8	5.68	0.77	5.97	35.1	5.88	2.2	0.7	5780	771
2½..............	29.4	6.27	0.82	5.97	32.8	5.48	2.4	0.3	4630	752

[a] Method of Test for Weight per Cubic Foot, Yield and Air Content (Gravimetric) of Concrete (C 138 – 44), 1958 Book of ASTM Standards, Part 4.

TABLE XII.—EFFECT OF MAXIMUM SIZE OF COARSE AGGREGATE ON STRENGTH OF CONCRETE (SERIES 163-A).

Each value average for 3 batches of concrete mixed on different days.

Sand Total Absolute Volume Aggregate, per cent	Fineness Modulus, Total Aggregate	b/b_o	Cement, sacks per cu yd	Water		Slump, in.	Air, per cent[a]	28-day Strengths, psi			
								Compression		Flexure	
				gal per cu yd	gal per sack			6 by 12 in. Cylinder	8 by 16 in. Cylinder	6 by 6 in. Beams	8 by 8 in. Beams
3/8-IN. MAXIMUM SIZE COARSE AGGREGATE											
50.6........	4.35	0.53	6.09	39.3	6.5	2.5	1.9	5600	5465	621	617
59.8........	4.05	0.42	6.03	41.7	6.9	2.6	3.0	4950	4760	567	547
69.0........	3.75	0.32	5.94	43.1	7.3	1.7	3.9	4360	4200	576	528
3/4-IN. MAXIMUM SIZE COARSE AGGREGATE											
31.3........	5.40	0.76	6.13	33.5	5.5	2.3	0.2	6265	6165	633	632
39.0........	5.10	0.66	6.05	34.5	5.7	2.6	1.6	6150	5935	615	614
46.8........	4.80	0.57	6.04	35.6	5.9	2.4	1.9	5900	5660	613	601
1½-IN. MAXIMUM SIZE COARSE AGGREGATE											
26.9........	6.02	0.82	6.09	31.2	5.1	1.8	−0.6	5835	5740	606	617
33.6........	5.72	0.73	6.05	31.9	5.3	2.6	0.4	5955	5810	625	605
40.3........	5.42	0.65	6.02	33.3	5.5	2.4	1.2	5960	5655	629	590
2½-IN. MAXIMUM SIZE COARSE AGGREGATE											
24.2........	6.55	0.82	6.07	29.9	4.9	2.3	−0.8	4465	4635	601	593
30.2........	6.25	0.74	6.03	30.4	5.0	2.5	0.1	5480	5275	586	580
36.2........	5.95	0.67	6.01	31.6	5.3	2.4	0.6	5640	5425	640	591

[a] See Table XI.

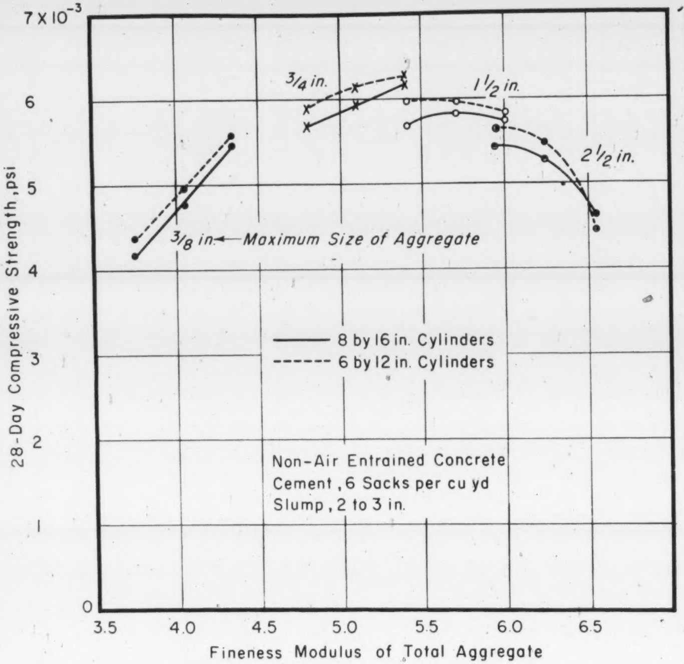

FIG. 6.—Relationship Between Fineness Modulus and Strength for Different Maximum Size Aggregates (Series 163-A).

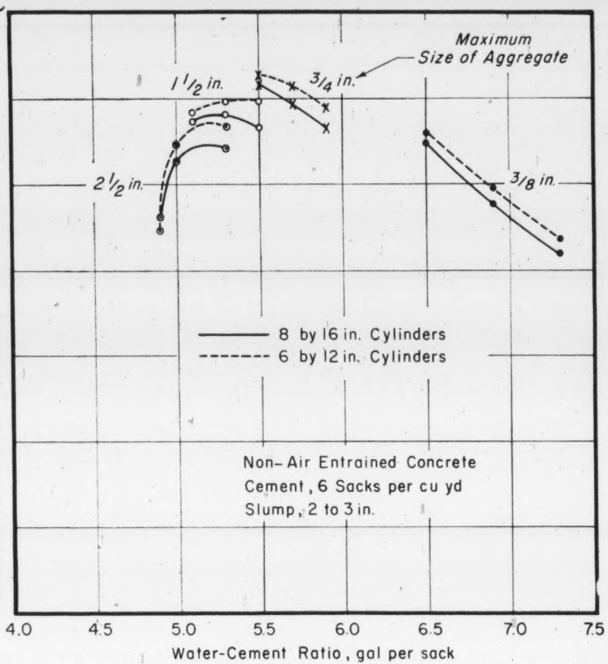

FIG. 7.—Relationships Between Water-Ratio and Strength for Different Maximum Size Aggregates (Series 163-A).

As was expected, lower strengths were secured for the larger specimens. On the other hand, the data provide no indication that size of specimen affected the relationship between strength and maximum size of aggregate found previously. As to sand content, it appears from Fig. 6 that the range in percentages might not have been large enough in all cases to produce the maximum strength obtainable. For the $\frac{3}{8}$-in. and $\frac{3}{4}$-in. maximum sizes, no mixes had sufficiently high fineness moduli (little enough sand) to cause a reduction in strength. For the $2\frac{1}{2}$-in. maximum size, fineness moduli were never low enough to give maximum strength. However, it is evident that these data support the previous finding of a decrease in strength with increasing maximum sizes larger than $\frac{3}{4}$ in.

TABLE XIII.—EFFECT OF MAXIMUM SIZE OF COARSE AGGREGATE ON COMPRESSIVE STRENGTH OF CONCRETE (SERIES J-112).

Each value average for either 4 or 5 batches of concrete mixed on different days. Compressive strengths averages for either 12 or 15 6 by 12 in. cylinders (3 from each batch).

Sand Total Absolute Volume Aggregate, per cent	Fineness Modulus, Total Aggregate	b/b_o	Cement, sacks per cu yd	Water		Slump, in.	Air, per cent[a]	28-day Compressive Strength, psi
				gal per cu yd	gal per sack			
$\frac{3}{8}$-IN. MAXIMUM SIZE COARSE AGGREGATE								
25	5.16	0.84	5.98	38.6	6.45	7.1	0.3	3360
30	4.99	0.71	6.00	39.1	6.51	5.3	0.5	4395
35	4.82	0.72	5.99	39.1	6.52	3.7	0.7	5165
40	4.65	0.66	6.00	39.3	6.55	4.8	1.2	5305
50	4.32	0.53	5.96	41.2	6.91	5.1	2.4	5150
75	3.47	0.24	5.98	49.1	8.21	4.8	3.8	3930
$\frac{3}{4}$-IN. MAXIMUM SIZE COARSE AGGREGATE								
25	5.61	0.83	6.03	35.2	5.84	5.1	−0.3	4130
30	5.42	0.77	6.03	35.6	5.90	4.3	0.0	4990
35	5.22	0.70	6.01	36.2	6.01	4.5	0.7	5600
40	5.02	0.64	6.01	37.0	6.15	4.5	1.2	5580
50	4.63	0.52	6.00	40.3	6.72	5.2	1.9	5195
75	3.62	0.24	6.01	48.6	8.09	4.4	3.9	3980
$1\frac{1}{2}$-IN. MAXIMUM SIZE COARSE AGGREGATE								
25	6.08	0.83	6.03	33.4	5.54	5.2	−0.3	4350
30	5.85	0.77	6.02	33.6	5.58	4.0	−0.1	5075
35	5.62	0.70	6.03	34.6	5.74	4.5	0.3	5425
40	5.39	0.64	6.02	36.0	5.98	4.7	0.8	5230
50	4.94	0.52	6.01	39.5	6.57	5.0	1.6	4915
75	3.78	0.23	5.99	48.4	8.08	4.8	3.8	3815
$2\frac{1}{2}$-IN. MAXIMUM SIZE COARSE AGGREGATE								
25	6.47	0.80	6.05	32.0	5.28	5.3	−0.4	4230
30	6.22	0.74	6.04	32.7	5.42	4.4	−0.2	4545
35	5.96	0.67	6.04	33.7	5.58	4.4	0.2	4895
40	5.71	0.62	6.04	35.4	5.86	4.3	0.5	5000
50	5.21	0.49	6.00	39.1	6.51	4.8	1.3	4705
75	3.91	0.22	6.02	48.6	8.08	4.4	3.3	3735

[a] See Table XI.

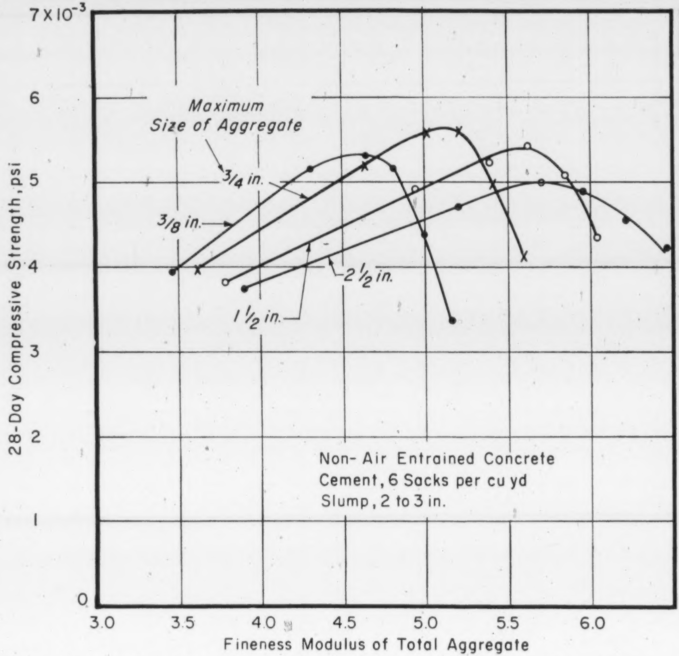

FIG. 8—Relationships Between Fineness Modulus and Strength for Different Maximum Size Aggregates (Series J-112).

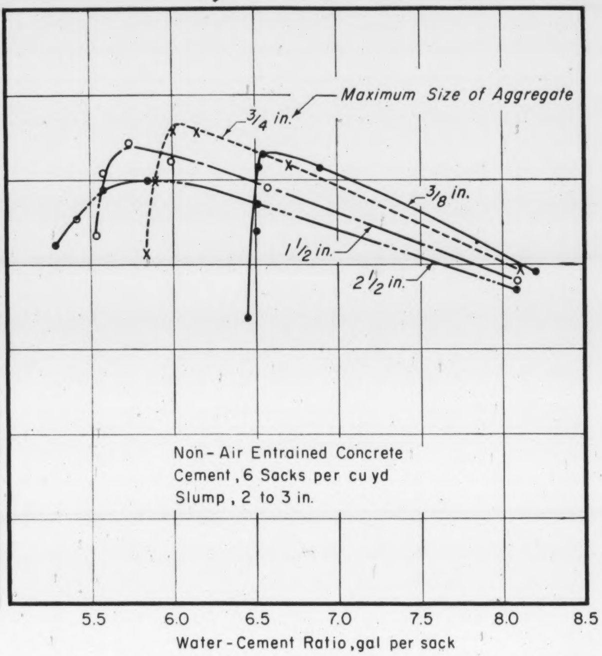

FIG. 9.—Relationships Between Water-Ratio and Strength for Different Maximum Size Aggregates (Series J-112).

The locations and shapes of the water-cement ratio curves in Fig. 7 also indicate less favorable strength development for the larger sizes of aggregate. It appears that, at a given water ratio, strength decreases progressively as maximum size of aggregate increases.

Comparisons Among Maximum Sizes of Coarse Aggregate for Wide Range of Sand Contents (Series J-112):

In spite of the convincing evidence from the investigations referred to, it was felt desirable to make at least one more group of tests in which there could be no doubt that results would include the optimum sand content for each coarse aggregate size. Tests were made with maximum sizes of $\frac{3}{8}$, $\frac{3}{4}$, $1\frac{1}{2}$, and $2\frac{1}{2}$ in., for one coarse aggregate only, using a cement factor of 6 sacks per cu yd and a nominal slump of 4 to 5 in. The-higher-than usual slump was used to minimize the likelihood of differences in compacting effects. Sand contents ranged from 25 to 75 per cent of the total aggregate for each maximum size. Compressive strength tests only were made on 6 by 12-in. cylinders. The data are summarized in Table XIII and the accompanying Figs. 8 and 9.

The results of these tests are clear-cut and unequivocal. They show that the mixing water demand decreased as maximum size of coarse aggregate increased, which is in accord with conventional thinking. On the other hand, they also show in clear-cut fashion that the compressive strength for the optimum fineness modulus of total aggregate (Fig. 8) or for the same water-cement ratio (Fig. 9) decreased as the maximum size increased.

Future Tests:

The results of these tests are so contrary to expectations and to interpretations of earlier data that some additional work is planned. Except in the case of series 155-B, all tests were made with the same aggregate, a very hard, high-quality, relatively rounded quartzite gravel. It is proposed to determine if something about the surface texture or high strength of particles of this gravel may have influenced the results. This seems unlikely in view of the fact that our findings appear to be in substantial agreement with those of the authors, where the tests were made with the extremely rough-textured blast-furnace slag.

MR. ELLIOT A. HALLER.[3]—With respect to the failure to follow preconceived results as to particle size of coarse aggregate, I have made some investigation of that phenomenon. It seems that by increasing the strength of the matrix up to a certain limit, that a point is reached where the weakest link of the chain is possibly the bond of the matrix to the coarse aggregate; therefore, if you have more cross-sectional surface area occupied by large-sized coarse aggregate, that may be the weakest link and may be one of the reasons why even a reduction in water does not necessarily result in higher flexural strength.

We know that, in polished aggregates or those with smooth surface texture, particularly gravel, we reach a plateau beyond which increasing the cement factor has no effect on increasing the flexural strength, which appears to be due to the lack of cementitious bond between the matrix and the coarse aggregate.

I wish to ask the author whether he used standard size specimens, as far as compressive strength and flexural strength, in all of his tests, and in cases where he used the 2-in. aggregate, whether the 2-in. aggregate was wet-screened from the mixture or whether it was tested with the 2-in. aggregate in both flexural and compressive strength specimens.

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[3] President, The Haller Testing Laboratories. Inc., New York, N. Y.

MR. D. W. LEWIS (*author*).—In reply to the question regarding the maximum size of the aggregate and the size of the specimens, we used standard 6 by 12-in. cylinders for compression, 6 by 6-in. cross-section beams for the flexural tests throughout. The 2-in. 1½-in. ¾-in., and ⅜-in. top sizes of aggregates were all used in the same size specimens in each case.

It is of some interest to compare the net water-cement ratios in these mixes with the various sizes of aggregate. They range from 5.2 gal per sack with 2-in. aggregate; 5.3 with the 1½-in. aggregate; 5.8 with the ¾-in., and went up to 6.4 with the ⅜-in. top size. So we had quite a large range in water-cement ratios.

MESSRS. D. W. LEWIS AND FRED HUBBARD (*authors' closure*).—The data submitted by Messrs. Walker, Bloem, and Gaynor are valuable in emphasizing the need for reexamination of some of the long-accepted relationships in the concrete field. In their discussion it is pointed out that increasing aggregate size beyond ¾ in. resulted in strength decreases for concretes with constant cement content and slump. All of their results are based on tests made after 28 days' curing and appear to be essentially in accord with our findings.

A careful study of the data in Table VIII of the paper, however, shows that the optimum aggregate size for maximum strength varies with length of curing in our tests. The concrete made with 2-in. top size slag aggregate had the highest compressive strength at 7 and 14 days; that made with 1½-in. aggregate was strongest at 28 and 90 days; while at one year the ¾-in. top size aggregate produced the strongest concrete. The same general trend is shown by the flexural strengths.

This indicates that, at least under some conditions, the optimum aggregate size may vary with the length of moist curing—or with the strength level of the concrete which is influenced by the curing. The curing time at which a given aggregate size would appear the best may vary greatly for different cement and aggregate characteristics. Thus while the tests reported by Messrs. Walker, Bloem, and Gaynor show optimum strength with ¾-in. aggregate after 28 days' curing, our results indicate the ¾-in. material to be best in compression only after one year of curing, but at a somewhat earlier age in flexure.

It is also possible, of course, that under some conditions a smaller size of aggregate may produce the higher strengths at early test ages. This is illustrated by the data in Table XIV, from tests recently completed in the National Slag Assn.'s laboratory. Although primarily a study of the effect of type of coarse aggregate, aggregate No. 13 (a crushed gravel) was used in both 1 and 1½-in. top sizes. The "standard" slag (No. 5) in the original test series was included for comparative purposes along with good quality aggregates of other types. The fine aggregate and all test procedures were identical to those used for the main series of tests. A new supply of type I-A cement was obtained from the same sources as used previously and blended in the same manner. All mixes were repeated on three different days except the standard mix (No. R5) which was repeated six times.

In these tests the 1-in. top size crushed gravel (mix No. 24) produced higher compressive strengths than did the 1½-in. top size (mix No. 23) at both the 7- and 28-day test ages. For flexure, the reverse is true, again indicating that the effects are not necessarily the same for the two types of test.

It seems probable that the optimum size of aggregate may be dependent upon a number of factors and vary not only with the surface texture and strength of aggregate particles as suggested by Messrs. Walker, Bloem, and Gaynor but also with strength characteristics of the cement, age at time of test, type of curing, etc. The many unanswered questions regarding this relationship are certainly worthy of further study.

The strength data shown in Table XIV indicate no significant differences in strength between the various types of aggregate tested. Whether the standard slag or the average for all aggregates is used as the basis of comparison, the percentage variations in strength of concrete made with the various aggregates is small.

TABLE XIV.—EFFECT OF TYPE OF COARSE AGGREGATE ON STRENGTH OF CONCRETE.

All concrete made with a blend of three sources of type I-A cement, 6 sacks per cu yd, 2 to 2½-in. slump, moist room cured until time of test. Except when otherwise indicated, top size of aggregate was 1½ in., and strengths shown are the averages for 3 tests in compression and 5 in flexure on 6 by 12-in. cylinders and 6 by 6-in. cross-section beams.

Mix	Coarse Aggregate				Compressive Strength, psi		Flexural Strength, psi	
		Type	Compact Weight per cubic foot, lb	Abrasion Loss, per cent	7 Days	28 Days	7 Days	28 Days
No. R5[a]........	No. 5	Slag	82.8	33.0	3620	4930	555	645
No. 19.........	No. 9	Limestone	103.6	25.0	3640	4990	565	630
No. 20.........	No. 10	Trap rock	106.0	10.5	3320	4520	565	645
No. 21.........	No. 11	Crushed gravel	101.0	21.1	3220	4280	575	645
No. 22.........	No. 12	Round gravel	111.0	19.9	3210	4400	500	630
No. 23.........	No. 13	Crushed gravel	106.2	24.0	3520	5060	510	625
No. 24[b]........	No. 13	Crushed gravel	103.2	24.0	3780	5300	505	590

[a] Strengths shown are averages for six tests in compression and ten in flexure.

[b] Top size of aggregate was 1 in.

The relative compressive and flexural strengths are of interest as compared to those obtained in the initial test series. Such a comparison can be made for slag aggregate No. 5 between the results shown for mix No. 5 in Table V and those for mix No. R5 in Table XIV. The greatest difference in flexural strength between the two test series for comparable test ages was 15 psi, representing a change of less than 3 per cent. In compression, however, the strengths obtained in the later tests were approximately 15 per cent lower than in the initial series.

The only variable to which this can be attributed is use of a new supply of cement (but from the same sources) in the test series reported in the accompanying Table XIV. The variation in the relationship of compressive to flexural strength with curing time was pointed out in the paper. It also appears that this relationship may change with different batches of cement from the same mills.

Since the flexural and compressive strengths react differently or to different degrees to many variables, careful investigation would be required before valid estimates of flexural strength could be made from compressive strength tests, a procedure that is currently being advocated because of inherent variability in the flexure test.

THE EFFECT OF TYPE OF CAPPING MATERIAL ON THE COMPRESSIVE STRENGTH OF CONCRETE CYLINDERS*

A REPORT OF THE DIVISION OF PHYSICAL RESEARCH, BUREAU OF PUBLIC ROADS

BY GEORGE WERNER[1]

In this investigation a study was made of different types of capping materials permitted for use on concrete specimens by current ASTM methods of test. The materials include portland cement, sulfur cement, and high-alumina cement. The use of high-strength gypsum plaster is permitted on concrete cylinders of low to moderate strength.

Early literature of the effect of capping materials on the compressive strength of concrete cylinders referred to sheet bedding materials or plaster of Paris. When it became apparent that these materials had an adverse effect on the strength of the test specimen, consideration was given to the use of other materials. Portland cement was of course a most logical substitute and its use was widely advocated. There was some dissatisfaction with the time required for the hardening of the portland cement, and attempts were made to accelerate the setting of the cement by the addition of plaster of Paris. Shortly thereafter, the use of sulfur cements was suggested and one of the earliest reports of this was by P. J. Freeman in 1928 (**6**).[2] Further modifications of capping materials were suggested and attempts to standardize capping procedures resulted in the requirements now given in ASTM Methods C 31 and C 192.[3] These standards not only specify acceptable capping materials but prescribe the planeness of bearing surfaces of test specimens. If the surfaces are not plane within 0.002 in., the specimens are required to be capped with neat portland cement, neat high-alumina cement, sulfur cement (a suitable mixture of sulfur and granular materials) or high-strength gypsum plaster. The caps must not fracture or flow under load and must have a minimum specified age before test. Gypsum plaster caps may not be used on specimens with expected strengths above 5000 psi.

Although it was believed that with the establishment of these ASTM standards, testing laboratories would adopt the practices and materials mentioned, it has been found that some laboratories still persist in using other capping practices. Consequently questions arise regarding the effect of these practices on the strength of concrete, and occasionally information is desired of the relative merits of the different capping materials permitted by the standards. Published data give little information of the effect of these materials when concrete cylinders of relatively low or high strength are tested. Nor are data found showing a direct comparison between the strengths of cylinders with rough or smooth ends after capping with materials other than hydraulic cements.

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[1] Highway Physical Research Engineer, Bureau of Public Roads, Division of Physical Research, Washington, D. C.

[2] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 1180.

[3] Method of Making and Curing Concrete Compression and Flexure Test Specimens in the Field (C 31 – 55), 1955 Book of ASTM Standards, Part 3, p. 1314.

Method of Making and Curing Concrete Compression and Flexure Test Specimens in the Laboratory (C 192 – 55), 1955 Book of ASTM Standards, Part 3, p. 1318.

SCOPE OF STUDY

Three series of tests were made by the Bureau of Public Roads to obtain information regarding the strengths of concrete cylinders, with either smooth or rough ends, when capped with various materials. The first series was made in 1949 on 6 by 12-in. concrete cylinders of high strength (7000 psi). One half of the number of cylinders tested were molded with smooth bottoms and tops and the remaining half had smooth bottoms and rough tops. The concrete was made with a type I cement, a natural sand of 2.75 fineness modulus, and a crushed limestone graded from $1\frac{1}{2}$ in. to No. 4. The second series was made in 1953 on 6 by 12-in. concrete cylinders of low strength (2000 psi) and high strength (7000 psi). The materials used were of the same type and grading as those in the first series of tests. One half of the cylinders were molded with smooth bottoms and tops and the remaining half had rough bottoms and tops.

The capping materials used were a neat high-alumina cement, a mixture of portland cement and plaster of Paris, two sulfur cements, two gypsum plasters, plaster of Paris, and sulfur without filler or additive. All capping materials used are commercially available.

When the results of these two series of tests were studied it was believed that the conclusions regarding the relative merits of caps of sulfur or sulfur cement might not apply to caps of the same materials but formed with a different type of capping device. Accordingly in 1956 a series of tests was made to determine the effect of the method of application of sulfur cement caps. In these tests, cylinders of high-strength concrete with smooth tops and bottoms were capped with a sulfur cement using both of the available methods, and the strengths obtained were compared with those of similar cylinders capped with high-alumina cement.

CONCLUSIONS

1. With one exception, cylinders capped with high-alumina cement gave higher crushing strengths for concretes of high and low strengths than were obtained with other capping materials such as gypsum plasters, plaster of Paris, a mixture of plaster of Paris and portland cement, sulfur cements, or sulfur alone.

2. Plaster of Paris caps or caps of a mixture of plaster of Paris and portland cement caused a large reduction (6 to 43 per cent) in the strengths of concrete cylinders of both classes when compared with similar cylinders capped with high-alumina cement.

3. Caps of high-strength gypsum plasters caused a decrease in the strength of cylinders of both classes of concrete as compared with similar cylinders capped with high-alumina cement. One type of gypsum plaster (type A) caused only a 3 to 5 per cent decrease.

4. Caps of one type of sulfur cement (type A), caused a reduction in the strength for cylinders of high-strength concrete, when capped horizontally, of 8 to 15 per cent below that of cylinders capped with high-alumina cement.

5. Caps of a second type of sulfur cement (type B), reduced the strength of cylinders of high-strength concrete capped horizontally approximately 20 to 28 per cent below that of similar cylinders capped with high-alumina cement.

6. Low-strength concrete cylinders were not affected to as great an extent as high-strength cylinders by the capping materials used. Low-strength cylinders capped with plaster of Paris or a mixture of plaster and portland cement had 6 to 11 per cent less strength than similar cylinders capped with high-alumina cement. Cylinders capped with other materials showed a loss in strength of 0 to 5 per cent.

7. Cylinders of high-strength concrete with rough ends showed lower strengths than similarly capped cylinders with smooth ends except for those capped with high-alumina cement or high-strength gypsum plaster. Caps of plaster of Paris or a mixture of plaster of Paris and portland cement caused the greatest reduction. For low-strength concrete, the strengths of cylinders with rough ends were approximately equal to strengths of similarly capped cylinders with smooth ends.

8. The thickness of a sulfur cement cap resulting from the method of application to the cylinder (vertical or horizontal) affected the resulting strength of high-strength concrete. Thick caps applied with the cylinder in a horizontal position caused a greater reduction in strength than thin caps applied with the cylinder in a vertical position.

GENERAL TEST PROCEDURE

The concrete cylinders were molded according to ASTM Method C 192[3] but variations were used for making rough tops or bottoms and for preventing loss of moisture. The top surface of the plastic concrete in the mold was roughened immediately after molding by making 10 indentations $\frac{1}{8}$ to $\frac{1}{4}$ in. deep with the finger tips. The bottom surfaces were made rough by casting the concrete against a hardened plaster of Paris insert in the mold. The insert had about 10 indentations of $\frac{1}{8}$ to $\frac{1}{4}$ in. in depth and was oiled before casting concrete against it to prevent sticking. The plaster insert was removed from the cylinder at an age of 20 to 24 hr after casting.

Smooth tops and bottoms of cylinders were made by molding the concrete against the oiled base plate and finishing the top surface of the plastic concrete with a small round-nosed pointing trowel. Most of the bottom surfaces were slightly outside of the required tolerance for planeness of 0.002 in. because indentations had been made in the metal base plates by the tamping rod. The surfaces of the smooth tops as trowelled varied from 0.1 in. to 0.2 in. from planeness.

The tops of cylinders of plastic concrete were not struck off flush with the tops of the cylinder molds as specified in ASTM Method C 192. All top concrete surfaces, either rough or smooth, were finished about $\frac{1}{8}$ in. below the top of the mold. This variation from standard procedure was used to prevent loss of water which might result if a glass plate were placed on the concrete flush with or slightly above the top of the mold and worked down until it touched the mold. However, a glass plate was used as a seal on the top of the metal mold after the cylinder had been molded and was covered immediately with wet burlap for 20 to 24 hr. The cylinders were removed from the molds at an age of 20 to 24 hr and stored in the moist room.

The cylinders tested in 1949 were made on one mixing day. Sixty-four cylinders of high-strength concrete were made in eight batches of eight cylinders per batch. One cylinder from each batch was tested for each of the eight variables of end condition and capping materials.

The cylinders tested in 1953 were made on six mixing days. Forty-eight cylinders of either high- or low-strength concrete were made per day in six batches of eight cylinders per batch. One cylinder from each batch was tested for each of

TABLE I.—PROPERTIES OF CAPPING MATERIALS—SERIES 1949.
Each value is the average of three or more tests except as noted.

Material	Type of Specimen, in.	Age at Test, hr	Compressive Strength, psi	Flexural Strength, psi	Sonic Modulus of Elasticity, psi
High-alumina cement (neat)	2 by 2 by 4 prism	24	14 000	...	...
	2 by 2 by 12 beam	24	...	1260	3.53×10^6
Plaster of Paris No. 1 molding	2 by 2 by 2 prism	3	1 680	...	...
	2 by 2 by 12 beam	3	...	385[a]	1.12
Type A gypsum plaster	2 by 2 by 2 prism	2	4 500	...	...
	2 by 2 by 12 beam	2	...	940[a]	2.49
Type B gypsum plaster	2 by 2 by 2 prism	2	3 460	...	...
	2 by 2 by 12 beam	2	...	795[a]	2.28
Type A sulfur cement	2 by 2 by 4 prism	2	7 850	...	...
	2 by 2 by 12 beam	[b]	...	1200	2.90

[a] Two tests.
[b] Tested at 17 days.

TABLE II.—PROPERTIES OF CAPPING MATERIALS—SERIES 1953.
Each value is the average of three or more tests.

Material	Type of Specimen, in.	Age at Test	Compressive Strength, psi	Flexural Strength, psi	Sonic Modulus of Elasticity, psi
High-alumina cement (neat)	2 by 2 by 12 beam	24 hr	...	1490	4.38×10^6
	3 by 6 cylinder	24 hr	8 930	...	...
	2 by 2 by 4 prism	9 days	17 030	...	...
Plaster of Paris No. 1 molding	2 by 2 by 12 beam	3 hr	...	...	0.804
	2 by 2 by 2 prism	3 hr	1 190	...	...
Plaster of Paris + portland cement, 50-50 by weight	2 by 2 by 12 beam	24 hr	...	...	1.32
	2 by 2 by 12 beam	48 hr	...	515	1.42
	2 by 2 by 2 prism	24 hr	1 420	...	...
	2 by 2 by 4 prism	48 hr	1 970	...	...
Type A gypsum plaster	2 by 2 by 12 beam	24 hr	...	805	2.12
	2 by 2 by 4 prism	24 hr	7 310	...	...
Type B gypsum plaster	2 by 2 by 12 beam	24 hr	...	790	2.12
	2 by 2 by 4 prism	24 hr	6 950	...	...
Type A sulfur cement	2 by 2 by 12 beam	3 hr	...	1180	3.48
	3 by 6 cylinder	3 hr	7 130	...	...
	2 by 2 by 4 prism	8 days	8 340	...	...
Type B sulfur cement	2 by 2 by 12 beam	3 hr	...	1295	2.31
	3 by 6 cylinder	3 hr	6 280	...	...
	2 by 2 by 4 prism	8 days	6 060	...	...
Sulfur 100 per cent	2 by 2 by 12 beam	3 hr	...	230[a]	1.46
	3 by 6 cylinder	3 hr	1 480	...	...
	2 by 2 by 4 prism	9 days	1 990	...	...

[a] Beams contained many cracks.

the eight variables of capping. Three batches of the concrete prepared each day were used for making cylinders with rough ends and three batches were used for cylinders with smooth ends.

TABLE III.—PROPERTIES OF CAPPING MATERIALS (1957).

Consistency of Paste	Round	Compressive Strength at Age Indicated, psi: Type A Gypsum Plaster (3 hr)[a]	Type B Gypsum Plaster (3 hr)[a]	Plaster of Paris (3 hr)[a]	50 per cent Plaster of Paris + 50 per cent Portland Cement (24 hr)[a]	High-Alumina Cement, First Lot, 24 hr[b]	High-Alumina Cement, Second Lot, 24 hr[c]
Very wet	1	4635	4500	1380	1445	8215	7875
	2	4700	4450	1370	1490	8060	7785
	3	4485	4315	1040	1450	9235	7875
	Average	4605	4420	1265	1460	...	7845
Optimum	1	5835	5035	2085	2000	8325	7675
	2	5935	5485	2125	2185	8085	8250
	3	5615	5200	1825	2250	10515	8500
	Average	5795	5240	2010	2145	...	8140
Very dry	1	6985	5835	2335	2265	9410	9650
	2	6750	5935	2390	2485	8980	9240
	3	6900	5900	2210	2590	13535	9660
	Average	6880	5890	2310	2445	...	9515

Each value is the average of tests on three 2-in. cubes.

[a] Specimens cured in laboratory air at 73 F and 50 per cent relative humidity.

[b] Specimens in rounds 1 and 2 cured (uncovered) in moist air at 73 F and 90 to 95 per cent relative humidity. Round 3 cured in moist air at 73 F and 90 to 95 per cent relative humidity but covered with glass plate and moist towel.

[c] Specimens cured (uncovered) in moist air at 73 F and 90 per cent relative humidity.

WATER CONTENTS, BY WEIGHT, OF CAPPING MATERIAL IN PASTE

Consistency of Paste	Type A Gypsum Plaster, per cent	Type B Gypsum Plaster, per cent	Plaster of Paris, per cent	50 per cent Plaster of Paris + Portland Cement, per cent	High-Alumina Cement, per cent
Very wet	30.9	31.4	56.0	50.2	34.4
Optimum	26.6	27.4	47.4	42.6	31.6
Very dry	22.8	22.9	41.8	39.2	26.8

All conditions of consistency of pastes would permit use for capping.

Cylinders of high-strength concrete that were tested in 1956 were made in three batches of nine cylinders each on each of three mixing days. Three cylinders from each batch were capped with high-alumina cement, another three were capped horizontally with one type of sulfur cement and the remaining three cylinders were capped vertically with the same type of sulfur cement.

CAPPING MATERIALS

The high-alumina hydraulic cement used in these tests has a high heat of hydration and requires careful moist curing. It develops compressive strengths at 24 hr in neat cement or mortar that equal or exceed the strengths obtained with type I portland cement at 28 days. When it is used for capping concrete cylinders, temperatures above 90 F of the paste or the surrounding air should be avoided. High temperatures or insufficient moisture for curing cause soft or "powdery" caps, and dry cylinders which absorb moisture from the neat cement paste cause high-alumina cement caps to shrink and crack.

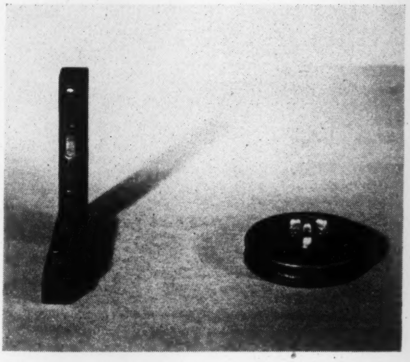

FIG. 1.—"L" Level and Two-Way Level.

The plaster of Paris used in these tests was an expensive type usually sold as No. 1 molding grade. It usually sets very rapidly. No retarder was used with it for capping and no difficulty was encountered in its use.

Type A and type B gypsum plasters are described by their manufacturer as extremely hard, low absorption plasters. The surface hardness and strength of type A castings are stated as seven to eight times that of ordinary molding plaster. Type B castings are described as having a surface hardness and tensile strength two to four times greater than standard plasters.

Sulfur cement caps were made of either of two commercial products, type A or type B, that are used for caulking pipe in water works or sewer construction. Each was melted in a metal pot on an electric hot plate. As no thermostatically controlled sand or oil bath was used around the pot, occasional overheating of the sulfur cement took place with a thickening of the cement and some vaporization of sulfur. On cooling to near the freezing point of sulfur, the cement became fluid again so that pouring the caps was not difficult. Reclaimed sulfur cement caps from the tested cylinders were reused.

Caps made of 100 per cent sulfur were applied in the same manner as the two sulfur cements for the horizontally capped cylinders. Overheating of the sulfur was avoided, and pouring of the molten material (without mineral filler) was easy because of its fluidity. The hardened caps had many fine cracks in the surface and no reclaimed sulfur was reused.

The properties of the capping materials as determined by tests on beams, cylinders, prisms, and cubes are given in Tables I, II, and III. With reference to the tests for compressive strength, the strongest material is the high-alumina cement. The two sulfur cements are the next strongest and are followed by the two gypsum plasters. Plain sulfur, the mixture of plaster of Paris and portland cement, and plain plaster of Paris are the three weakest materials. All materials having hydraulic properties appear to be affected to a marked extent by variations in the amount of water used to prepare them.

CAPPING PROCEDURES

Caps of high-alumina cement were applied usually within the first three days after the cylinders had been removed from the molds. One end was capped on one day and the other end was capped on the following day. Plate glass 7 by 7 by $\frac{1}{4}$ in. thick with surfaces within

0.001 in. of planeness was used to form the bearing surfaces of the caps. The plates were coated with a thin film of graphite grease before use. Each cylinder was checked with an "L" level and a two-way level during capping to secure caps with parallel surfaces perpendicular to the axis of the cylinder. These levels are shown in Fig. 1. All capping with high-alumina cement was done in the moist room where a temperature of 73 ± 2 F and a relative humidity of 98 to 100 per cent were maintained.

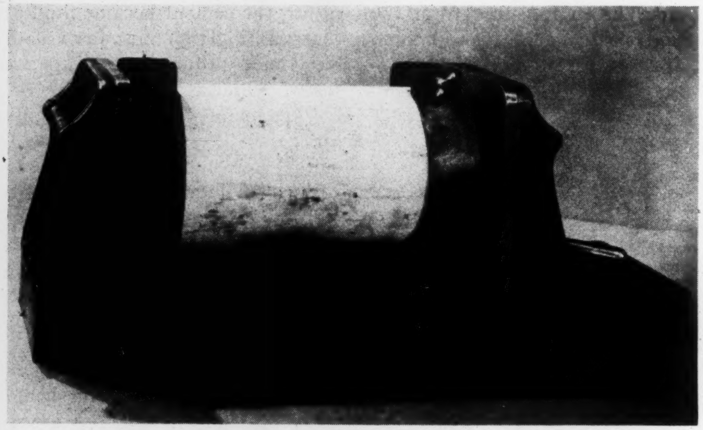

FIG. 2.—Nardiello Horizontal Capping Device for Sulfur Cement.

Caps of plaster of Paris or gypsum plasters were applied to cylinders two to three hours before testing by a procedure identical with that used for the high-alumina cement caps. As capping was done in laboratory air of 65 to 75 F temperature with a variable humidity of 20 to 50 per cent, the cylinders were wrapped in wet burlap between the caps to prevent loss of moisture.

Caps of a mixture of plaster of Paris and portland cement were applied to cylinders 24 hr prior to testing. The capping procedure was identical with that used for caps of gypsum plasters or of plaster of Paris alone. After being capped, the cylinders were wrapped in wet burlap and stored in laboratory air.

Caps of sulfur cement, type A or type B, or sulfur alone were applied to cylinders tested in 1949 and 1953 by a horizontal Nardiello type capping device shown in Fig. 2. Caps of the type A sulfur

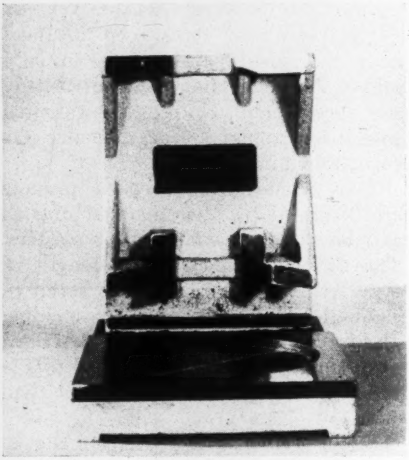

FIG. 3.—Vertical Capping Device for Sulfur Cement.

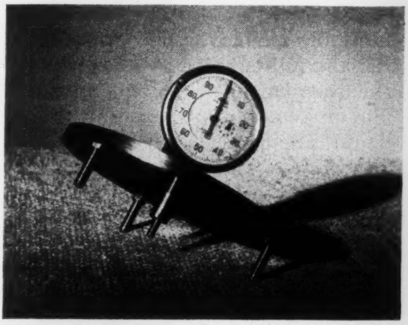

FIG. 4.—Surface Gage for Checking Caps, 1953 Series.

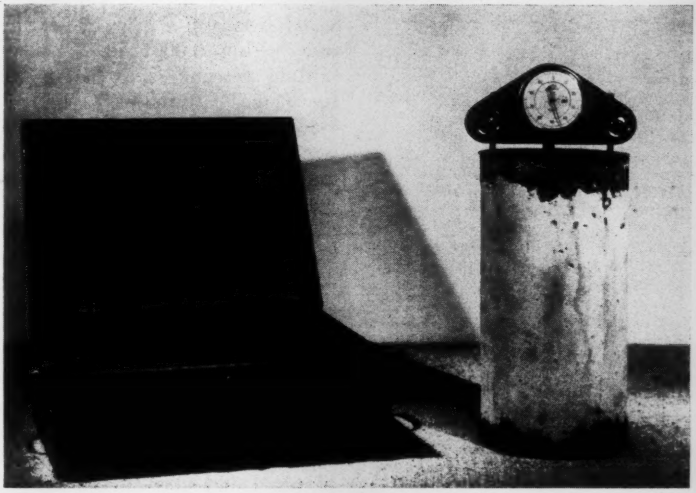

FIG. 5.—Surface Gage and Surface Plate for Checking Caps, 1956 Series.

cement used on the cylinders tested in 1956 were applied by this device or by a vertical type of device, shown in Fig. 3, in which the end of a cylinder is set in a "puddle" of the molten material. After being capped, the cylinders were wrapped in wet burlap until tested two or more hours later.

Metal plates on the two types of capping devices for sulfur or sulfur cements were within 0.0005 in. of planeness. They were oiled with a thin film of petroleum oil before use, but care was taken to avoid an excess that might affect the quality of the sulfur or sulfur cement. Oil was not used on the cylinder ends to facilitate recovery of sulfur cement caps.

Caps of neat alumina cement, plaster of Paris, gypsum plasters, or a mixture of plaster and portland cement varied in average thickness from $\frac{1}{8}$ in. for specimens with smooth ends to $\frac{1}{4}$ in. for specimens with rough ends. Depressions in the rough ends of cylinders were filled with capping material before placing the cylinder vertically on the plastic capping material.

Caps of type A and type B sulfur cements and sulfur alone varied from $\frac{1}{4}$ in. for smooth end cylinders to $\frac{5}{16}$ in. for rough end cylinders when the Nardiello type of capping device was used. The thickness of caps made with this horizontal capping device is governed by the minimum opening of the ring at the end

TABLE IV.—COMPRESSIVE STRENGTHS OF HIGH-STRENGTH CONCRETE—6 BY 12-IN. CYLINDERS—1949 SERIES.

Capped[b] With....	High-Alumina Cement, psi	Type A Gypsum Plaster, psi	Type A Sulfur Cement, psi	Plaster of Paris, psi
Rough Cylinder Tops,[a] Smooth Bottoms				
Batch:				
No. 1	7080	6560	6680	4630
No. 2	6880	6580	6970	4180
No. 3	6990	6390	6540	4600
No. 4	7230	6900	7050	4560
No. 5	7140	7030	6580	4550
No. 6	7090	7360	7020	4740
No. 7	6630	6510	6300	5400
No. 8	6970	7070	6700	5090
Average	7000	6800	6730	4720
Average variation, per cent	1.9	4.3	3.2	5.7
Maximum variation, per cent	5.3	8.2	6.4	14.4
Coefficient of variation, per cent[c]	2.5	4.7	3.7	7.4
Smooth Cylinder Tops, Smooth Bottoms				
Batch:				
No. 1	7190	6870	6660	5800
No. 2	6930	6720	6060	6080
No. 3	6970	6970	6810	6040
No. 4	7100	6950	6010	6370
No. 5	6860	6460	6500	6100
No. 6	6870	7270	6890	6520
No. 7	6690	6810	6330	5200
No. 8	6530	6710	6040	5920
Average	6890	6840	6410	6000
Average variation, per cent	2.3	2.5	4.7	4.6
Maximum variation, per cent	5.2	6.3	7.5	13.3
Coefficient of variation, per cent[c]	2.9	3.2	5.2	6.2

All cylinders were continuously moist cured and tested at 25 days.

[a] Depressions in rough tops were 1/8 to 1/4 in. Smooth tops were trowelled and smooth bottoms were 0.002 to 0.004 in. out of planeness, prior to capping.

[b] Age of caps at time of test:

High-alumina cement	24 hr +
Plaster of Paris	3 hr
Type A gypsum plaster and type A sulfur cement	2 to 3 hr

[c] Quality Control of Materials (*ASTM STP No. 15C*).

of the cylinder that will be sufficient to permit the molten capping material to enter and fill the space completely without entrapping air by too rapid cooling.

Sulfur cement caps made with the vertical capping device used in the 1956 series of tests varied in average thickness from $\frac{1}{16}$ to $\frac{1}{8}$ in. In many cases, air voids were found between the cylinder and cap because air was trapped in the molten sulfur cement when the end of a cylinder was placed therein.

All cylinders were tested in a hydraulic testing machine of 400,000-lb capacity. The face of the upper spherical bearing block was within 0.0004 in. of planeness and the face of the lower bearing block was within 0.0001 in. The top and bottom bearing faces had a Rockwell hardness of C 60+. Cylinders were loaded at the rate of 55,000 lb (total load) per min, and the controls of the testing machine were not adjusted while a cylinder was yielding rapidly immediately before failure.

Prior to making tests for compressive strength in the 1953 series, the surface of each capped end of a cylinder was checked with a surface gage shown in Fig. 4. Another type of gage and surface plate used to check the planeness caps on cylinders tested in the 1956 series is shown in Fig. 5. The surface plate is made of granite and is plane within 0.0001 in.

RESULTS OF TESTS FOR COMPRESSIVE STRENGTH

The results of compressive strength tests made in 1949 on concrete cylinders of high-strength concrete are shown in Table IV. Results of tests made in 1953 are shown in Tables V and VI for high- and low-strength concrete respectively. The results of tests made in 1956 to determine the effect of the method used in capping cylinders with a sulfur cement are given in Table VII. These last tests were made only on high-strength concrete.

TABLE V.—COMPRESSIVE STRENGTHS OF HIGH-STRENGTH CONCRETE—6 BY 12-IN. CYLINDERS—1953 SERIES.

Cylinder Ends Before Capping	Batch and Round Numbers	High-Alumina Cement Caps, psi	Type A Gypsum Plaster Caps, psi	Type B Gypsum Plaster Caps, psi	Plaster of Paris Caps, psi	Plaster + Portland Cement Caps, psi	Type A Sulfur Cement Caps, psi	Type B Sulfur Cement Caps, psi	Sulfur 100 per cent Caps, psi
Rough.........	Nos. 1-1	7820[a]	7250	6290	3970	4070	6340	6390[b]	4610[a]
Rough.........	Nos. 2-1	7600[a]	7270	5970[a]	4350	4090[a]	6990	5870[a]	5160
Rough.........	Nos. 3-1	7230	7820	6170	4410	3080	6950[a]	6310	5680
Rough.........	Nos. 1-2	7450	7270	6210[a]	3920	4690	6430	4830	6270[a]
Rough.........	Nos. 2-2	7690	7400	5920	4710	4690	6030[a]	4140[b]	4790[a]
Rough.........	Nos. 3-2	7470	7200	5920[a]	3740	4420	6180[a]	4700[b]	4920[a]
Rough.........	Nos. 1-3	7720	6850	5410	4280	4850	6350[a]	4870[b]	5470
Rough.........	Nos. 2-3	7850	6790	5500	4730	4910	6370	5810[b]	5630
Rough.........	Nos. 3-3	7420	6860	5280	4600	5310	6350	5980[b]	5270
Average.................		7580	7190	5850	4300	4460	6440	5430	5310
Average variation, per cent...		2.3	3.3	7.7	6.7	10.8	3.6	13.1	7.6
Maximum variation, per cent.		4.6	8.8	9.7	10.0	30.9	8.5	23.8	18.1
Coefficient of variation, per cent		2.6	4.3	5.9	7.8	13.7	4.7	14.0	9.2
Smooth........	Nos. 4-1	6690	6490	6370	5770	5090	6450	6020[a]	5840
Smooth........	Nos. 5-1	7570	6950	6620	5490	4990	6550	6010[a]	5980
Smooth........	Nos. 6-1	7480	6620	6680	5430	5840	6500	5940[a]	5540
Smooth........	Nos. 4-2	6200	7010	6330	6060	5870	6270	5210[a]	5980
Smooth........	Nos. 5-2	7390	7050	6640	6120	5080	6890	5750[a]	6390[a]
Smooth........	Nos. 6-2	6990	6830	6490[a]	4650	5060	6660[a]	5570[a]	5290
Smooth........	Nos. 4-3	7360	6990	6380[a]	5470	5400	6640	6010[a]	6140
Smooth........	Nos. 5-3	7160	6760[a]	6190	5140	5950	6420[a]	6150[a]	5580
Smooth........	Nos. 6-3	7400	7290	6160	5470	5640	6560	4880	6580
Average.................		7140	6890	6430	5510	5440	6550	5730	5920
Average variation, per cent...		4.8	2.7	2.5	5.7	6.4	1.9	5.9	5.4
Maximum variation, per cent.		13.2	5.8	4.2	15.6	9.4	5.2	14.8	11.1
Coefficient of variation, per cent		5.9	3.3	2.8	7.7	6.8	2.5	7.1	6.6

Cylinders were continuously moist cured and tested at 28 days.

Depressions in rough ends of cylinders were ⅛ to ¼ in. Smooth tops of cylinders were trowelled and smooth bottoms were 0.002 to 0.004 in. out of planeness.

Age of caps at time of test:

High-alumina cement..	24 hr +
Plaster + portland cement—24 hr, plaster of Paris..........................	3 hr
Types A and B gypsum plasters and types A and B sulfur cements, sulfur........	2 to 3 hr

[a] One cap of cylinder 0.0015+ out of planeness.

[b] Two caps of cylinder 0.0015+ out of planeness.

In each series of tests, the uniformity of the compressive strengths as affected by the use of each capping material or the end condition of the cylinders was calculated. On the assumption that a coefficient of variation (see ASTM Manual on Quality Control, *ASTM STP No. 15C*, and Recommended Practice for Evaluation of Compression Tests Results of Field Concrete, *Journal*, Am. Concrete Inst., July, 1957, Vol. 1, No. 29) of 7 per cent or less indicates satisfactory duplication of test results for specimens made, cured, capped, and tested in the laboratory, the results obtained with each capping material were appraised for

TABLE VI.—COMPRESSIVE STRENGTHS OF LOW-STRENGTH CONCRETE—6 BY 12-IN. CYLINDERS—1953 SERIES.

Cylinder Ends Before Capping	Batch and Round Numbers	High-Alumina Cement Caps, psi	Type A Gypsum Plaster Caps, psi	Type B Gypsum Plaster Caps, psi	Plaster of Paris Caps, psi	Plaster + Portland Cement Caps, psi	Type A Sulfur Cement Caps, psi	Type B Sulfur Cement Caps, psi	Sulfur 100 per cent Caps, psi
Rough	Nos. 1-1	2190	2030	2140[a]	1840	2130	2330[a]	2150[b]	2240[b]
Rough	Nos. 2-1	2160	2260	2030[a]	2110	2070	2360	2310[b]	2150[b]
Rough	Nos. 3-1	2190	2040	2000	1980	1850	2050	2090[b]	1930[a]
Rough	Nos. 1-2	2120	2330	2130	1890	2200	2160[a]	2060[a]	2090[b]
Rough	Nos. 2-2	2290	2090	2140	2020	2320	2360	2200	2270[a]
Rough	Nos. 3-2	2340	2190	2120	1960	2260	2080[a]	2080	2050[a]
Rough	Nos. 1-3	2120	2150	2150[a]	2040	1960	2340	2250[a]	2190[b]
Rough	Nos. 2-3	2370	2360	2340	2250	2070	2570[a]	2380[a]	2310[b]
Rough	Nos. 3-3	2250	2020	2240	1870	1930	2370[b]	2370[a]	2190[b]
Average		2230	2160	2140	2000	2090	2290	2210	2160
Average variation, per cent		3.5	5.0	3.1	4.9	6.0	5.7	4.7	4.2
Maximum variation, per cent		6.3	9.3	9.3	12.5	11.5	12.2	7.7	10.6
Coefficient of variation, per cent		3.9	5.7	4.5	6.1	7.1	6.8	5.3	5.2
Smooth	Nos. 4-1	1950	1800	1800	1780	1860	1920	2020	1960[b]
Smooth	Nos. 5-1	2130	2060	2060[b]	1920	1850	1850	2010	1860[b]
Smooth	Nos. 6-1	2300	2020	2160[a]	1820	1930	2140	2040	2150[b]
Smooth	Nos. 4-2	2210	2110	2170[a]	1990	2140	1930	2020[b]	2260[b]
Smooth	Nos. 5-2	2240	2230	2120	2060	1950	2090	2090	1900[a]
Smooth	Nos. 6-2	2140	2010	1970[b]	2070	2180	1950	1990	1940[b]
Smooth	Nos. 4-3	2230	2380	2230	2080	2230	2400	2430[a]	2190[b]
Smooth	Nos. 5-3	2490	2140	2310	2010	2180[a]	2440	2230[a]	2500[a]
Smooth	Nos. 6-3	2160	2260	2230[b]	1920	1920	2160	2220	2090[a]
Average		2210	2110	2120	1960	2030	2100	2120	2090
Average variation, per cent		4.4	5.9	5.4	4.6	6.8	7.9	5.5	7.7
Maximum variation, per cent		12.7	14.7	15.1	9.2	9.9	16.2	14.6	19.6
Coefficient of variation, per cent		6.2	7.5	6.9	5.3	7.1	9.5	6.5	9.4

Cylinders were continuously moist cured and tested at 28 days.

Depressions in rough ends of cylinders were ⅛ to ¼ in. Smooth tops of cylinders were trowelled and smooth bottoms were 0.002 to 0.004 in. out of planeness.

Age of caps at time of test:

High-alumina cement	24 hr+
Plaster + portland cement—24 hr, plaster of Paris	3 hr
Types A and B gypsum plasters and types A and B sulfur cements, sulfur	2 to 3 hr

[a] One cap of cylinder 0.0015+ out of planeness.

[b] Two caps of cylinder 0.0015+ out of planeness.

uniformity. In the 1949 series, only the cylinders of high-strength concrete, with rough tops, and capped with plaster of Paris had coefficients of variation in excess of 7 per cent. In the 1953 series, a number of individual sets of specimens showed a high coefficient of variation, but those tested in 1956 had satisfactory reproducibility. To summarize these results with respect to capping material only, the following values are given for all sets of specimens. These values refer to the number of sets of specimens capped with a single material and

TABLE VII.—COMPRESSIVE STRENGTHS OF HIGH-STRENGTH CONCRETE—6 BY 12-IN. CONCRETE CYLINDERS—1956 SERIES.

Mixing Days	Batch	Smooth Cylinder Tops and Bottoms[a] Capped with		
		High-Alumina Cement, psi	Type A Sulfur Cement Capped Horizontally, psi	Type A Sulfur Cement Capped Vertically, psi
1.......	No. 1	6130	5910	5830
		6310	5910	6090
		6030	5970	6140
1.......	No. 2	6560	6180	6410
		6370	6130	6170
		6490	6190	6340
1.......	No. 3	6480	5750	6410
		6380	6080	6340
		6490	6120	6140
2.......	No. 1	6370	6140	6250
		6370	5910	6390
		6170	5800	6270
2.......	No. 2	5990	5900	6040
		6270	6060	5900
		6050	5870	5480[c]
2.......	No. 3	6320	5270[b]	6460
		6390	5360[b]	6200
		6290	6220	6540
3.......	No. 1	6020	5450	5970
		5850	5340	5660
		6080	5470	5930
3.......	No. 2	5990	5940	6150
		6340	5070	5880
		5870	5050	5910
3.......	No. 3	6150	5790	5910
		6100	5420	5950
		6340	5960	5960
Average.......		6230	5830	6120
Average variation, per cent.......		2.8	4.5	3.2
Maximum variation, per cent...		6.1	13.4	7.5
Coefficient of variation, per cent...........		3.1	5.7	3.7

Cylinders were continuously moist cured and tested at 28 days.

Age of caps at time of test = 2 to 3 hr for sulfur cement, 24 hr + for high alumina cement.

[a] Smooth tops were trowelled, bottoms of cylinders were 0.002 to 0.004 in. out of planeness before capping. Age of sulfur cement caps at testing was 2 to 3 hr.

[b] Not averaged, cylinders split on testing.

[c] Not averaged, cylinder cap flowed.

showing a coefficient of variation in excess of 7.0 per cent:

High-alumina cement: 0 of 7 sets
Type B gypsum plaster: 0 of 5 sets
Type A gypsum plaster: 1 of 6 sets
Type A sulfur cement: 1 of 7 sets
Type B sulfur cement: 2 of 4 sets
Sulfur: 2 of 4 sets
Plaster of Paris: 3 of 6 sets
Plaster and portland cement: 3 of 4 sets

From the above, it appears that it may be more difficult to obtain uniform strengths with specimens capped with plaster of Paris, a combination of plaster of Paris and portland cement, type B sulfur cement, and sulfur than with the other capping materials. Since all of the caps were prepared by experienced laboratory technicians, it is believed that the reasons for the greater variability of the specimens capped with these four materials lie in the characteristics of the materials such as thermal or moisture volume change and rate of setting. It will be observed that many of the caps of type B sulfur cement or sulfur on the specimens tested in 1953 were over 0.0015 in. from planeness. Since some caps that were nearer to planeness were prepared with these materials and since all caps were prepared using the same equipment, it is suggested that a high thermal volume change of some materials containing sulfur may prevent the attainment of satisfactory caps as is possible with other materials.

The principal items of interest in these tests are the very marked differences between the strengths of specimens of high-strength concrete when capped with different materials and the smaller differences between the strengths of specimens of low-strength concrete when capped with the same materials. Tables VIII and IX and Figs. 6 and 7 present summaries of the results of this investigation. With respect to high-strength concrete, it is seen that specimens capped with high-alumina cement, type A high-strength gypsum plaster, and type A sulfur cement (applied with

TABLE VIII.—SUMMARY OF STRENGTH RESULTS.

Capping Material	Average Compressive Strength of Cylinders, psi			
	High Strength Concrete		Low Strength Concrete	
	Rough Ends	Smooth Ends	Rough Ends	Smooth Ends
1949 tests				
High-alumina cement	7000	6890	...	...
Gypsum plaster, type A	6800	6840	...	...
Gypsum plaster, type B	...	6410	...	...
Sulfur cement, type A	6730	...	...	...
Plaster of Paris	4720	6000	...	...
1953 tests				
High-alumina cement	7580	7140	2230	2210
Gypsum plaster, type A	7190	6890	2160	2110
Gypsum plaster, type B	5850	6430	2140	2120
Plaster of Paris	4300	5510	2000	1960
Plaster of Paris + cement	4460	5440	2090	2030
Sulfur cement, type A[a]	6440	6550	2290	2100
Sulfur cement, type B[a]	5430	5730	2210	2120
Sulfur, 100 per cent	5310	5920	2160	2090
1956				
High-alumina cement	...	6230	...	...
Sulfur cement, type A[a]	...	5830	...	...
Sulfur cement, type A[b]	...	6120	...	...

[a] Capped horizontally in Nardiello device.

[b] Capped vertically.

TABLE IX.—RELATION OF STRENGTH RESULTS.

Capping Material	Reduction in Average Strengths of Cylinders				Ratio of Strengths of Cylinders	
	High Strength Concrete		Low Strength Concrete		Smooth Ends Rough Ends	
	Rough Ends, per cent	Smooth Ends, per cent	Rough Ends, per cent	Smooth Ends, per cent	Low Strength Concrete	High Strength Concrete
1953 tests						
High-alumina cement	0	0	0	0	0.94	0.99
Gypsum plaster, type A	5.1	3.5	3.1	4.5	0.96	0.98
Gypsum plaster, type B	22.8	9.9	4.0	4.1	1.10	0.99
Plaster of Paris	43.3	22.8	10.3	11.3	1.28	0.98
Plaster of Paris + portland cement	41.2	23.8	6.3	8.1	1.22	0.97
Sulfur cement, type A (caps applied horizontally)	15.0	8.3	+2.7	5.0	1.02	0.92
Sulfur cement, type B (caps applied horizontally)	28.4	19.7	0.9	4.1	1.06	0.96
Sulfur	29.9	17.1	3.1	5.4	1.11	0.97
1956 tests						
High-alumina cement	...	0	...	...	...	...
Sulfur cement, type A (caps applied horizontally)	...	6.4	...	...	...	...
Sulfur cement, type A (caps applied vertically)	...	1.8	...	...	...	

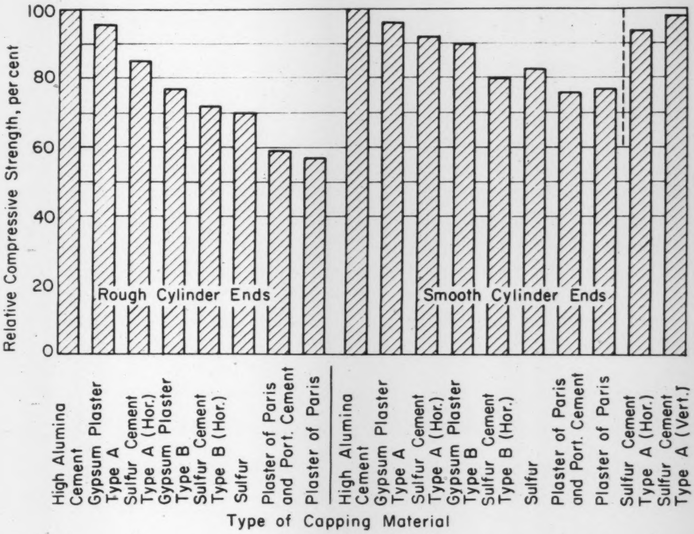

FIG. 6.—Relative Strengths of High Strength Concrete Cylinders (1953 plus 1956 Series).

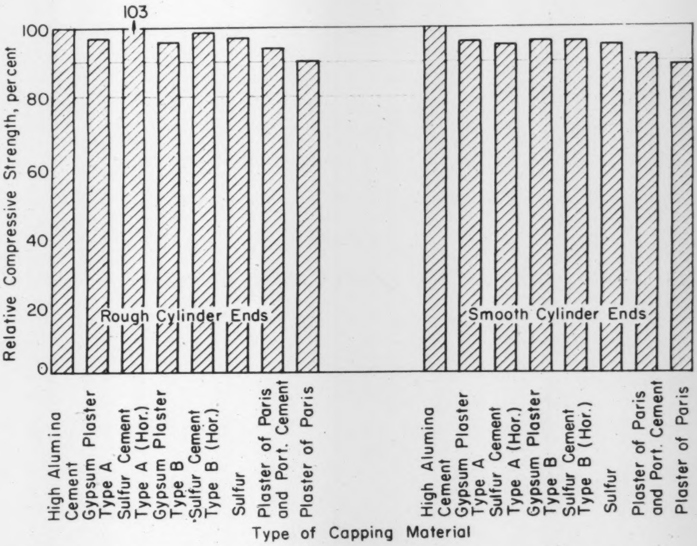

FIG. 7.—Relative Strengths of Low Strength Concrete Cylinders (1953 Series).

the specimens in a vertical position) gave essentially the same compressive strength. Specimens capped with type B sulfur cement, sulfur, plaster of Paris or a mixture of plaster of Paris and portland cement gave strengths from 20 to 43 per cent below those for the specimens capped with high-alumina cement.

Greatly different results were obtained in the tests of the specimens of low-strength concrete. The lowest strengths obtained were only 11 per cent less than the standard strengths of similar cylinders with caps of high-alumina cement. Specimens prepared with five of the seven capping materials differed in strength by only 5 per cent or less from the standard strength.

The results reported here show that the choice of capping material for concrete cylinders is of great importance when high-strength concrete is tested. In these tests, the high-strength concrete had a strength of 7000 psi, but it is expected that specimens having strengths of 5000 psi or more would show a similar behavior.

With high-strength concrete, the use of caps prepared from high-alumina cement, type A high-strength gypsum plaster, or type A sulfur cement is found desirable. In the case of the type A sulfur cement, the caps should be applied to the specimen in a vertical position.

A reason for the marked differences between the strengths of specimens of high-strength concrete capped with such similar materials as the two gypsum plasters or the two sulfur cements is not known. The results of tests on the capping materials given in Tables II and III do show that the type A gypsum plaster is stronger than the type B gypsum plaster, and in Table II that the type A sulfur cement is stronger in compression than the type B sulfur cement. It appears that it would be very desirable for the capping material to have a compressive strength appreciably higher than that of the concrete specimen as is obtained by the use of neat high-alumina or neat portland cement paste. And it also appears that with concrete of higher strength than 5000 psi becoming common, the provisions for capping as given in ASTM Method C 192 might be revised to require a minimum compressive strength for sulfur cements, and to emphasize more strongly the need for ample time between capping and testing to permit the capping material to gain its maximum strength.

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(16) S. Ahmed, "Effect of Capping on the Compressive Strength of Concrete Cubes," *Magazine of Concrete Research* (Great Britain), March 1955.

DISCUSSION

MR. S. B. HELMS[1] (*presented in written form*).—Good capping materials and testing practices are essential in making compressive strength tests of concrete according to ASTM Method C 39.[2] Certainly we are all indebted to Mr. Werner and the Bureau of Public Roads for this comprehensive report of what seems to be a continuing investigation of methods of capping specimens. The capping provisions now in ASTM Methods C 31 and C 192,[3] for making strength test specimens, were incorporated as revisions in 1949 after the results of the Bureau of Public Roads 1949 series were made available to Committee C-9. Since that time only minor changes have been made to clarify the intent of these provisions. At present the specifications permit only sulfur mix caps, or high-strength gypsum caps, to be applied to concrete cylinders less than 18 hr before testing.

The author lists G. E. Troxell's paper as a reference[4] and the accompanying Table X is a reproduction of tabular summary from E. N. Vidal's (**1**)[5] discussion of the Troxell paper. Each value is an average strength for sixty 6 by 12-in. concrete cylinders tested at ages of 7, 28, and 90 days. Additional details of these tests are given in Table 64 of *Bulletin 4*, "Mass Concrete Investigations"—Boulder Canyon Project (**2**). Some advantages in using sulfur mixtures for capping (provided suitable melting and ventilating equipment is employed) are convenience, speed, and a negligible effect on the moisture content of the specimen. Vidal pointed out that an economical sulfur mixture prepared in the laboratory would eliminate the need for salvaging the capping mixture. This fact has been stated more recently by McDonell (**3**) in February 1955 issue of *ACI Journal.* Practices of salvage and reuse of sulfur mixture should be investigated and evaluated. Reheating salvaged sulfur cement which contains a plasticizer may result in inferior caps if contaminated by oil.

Tests of specimens with ends ground plane provides a standard of comparison which has been adopted in some of the

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[1] Research Engineer, Lehigh Portland Cement Co., Research Laboratory, Allentown, Pa.

[2] Tentative Method of Test for Compressive Strength of Molded Concrete Cylinders (C 39 – 56 T), 1958 Book of ASTM Standards, Part 4.

[3] Method of Making and Curing Concrete Compression and Flexure Test Specimens in the Field (C 31 – 57), 1958 Book of ASTM Standards, Part 4.

Method of Making and Curing Concrete Compression and Flexure Test Specimens in the Laboratory (C 192 – 57), 1958 Book of ASTM Standards, Part 4.

[4] See reference (**13**) of the author's paper.

[5] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 1186.

TABLE X.—EFFECT OF END CONDITIONS ON STRENGTH OF 6 BY 12 IN. CYLINDERS.

Results presented in Vidal discussion of Troxell paper.[a]

Capping Method	Age of Cap	Compressive Strength, psi	Relative Strength	Coefficient of Variation, per cent
Ends ground plane	...	4030	100	6.2
Portland cement[b]	...	4030	100	7.1
Sulfur mix	30 min	4070	101	5.7
Lumnite cement	24 hr	4090	101.5	5.5
Plaster	4 hr	3890	97	6.4
Plaster-cement	4 hr	3930	97.5	6.1

[a] *Proceedings*, Am. Soc. Testing Mats., Vol. 41, p. 1046 (1941).

[b] Capped 2 hr after molding cylinder.

tables in this discussion. The sulfur mix capping material used in our laboratory was described in a brief article by H. G. Collins (4) which appeared in the June 1941 *ACI Journal*. Our laboratory-prepared mixture consists of 6 parts sulfur, 3 parts fine banding sand, and 1 part fly ash melted at about 275 F. Since 1941, when this article and the Vidal discussion were published, we have been applying relatively thin sulfur mix caps by means of a vertical capping device in our laboratory.

The advantages of thin caps are well known. Table VII in the author's paper points to the advantages of sulfur mix caps applied using the "vertical" capping technique. This is particularly true for high-strength concrete specimens. Figure 8 is a diagrammatic interpretation of test data obtained at our laboratory by A. L. Bowman submitted to interested members of Committee C-9 in 1954. These curves were drawn by inspection through the plotted points of individual specimens; it is of interest that there was practically no scatter from the solid line representing Vitrobond. This chart indicates that cap about $\frac{1}{8}$-in. thick made with these materials would be satisfactory for high-strength concrete.

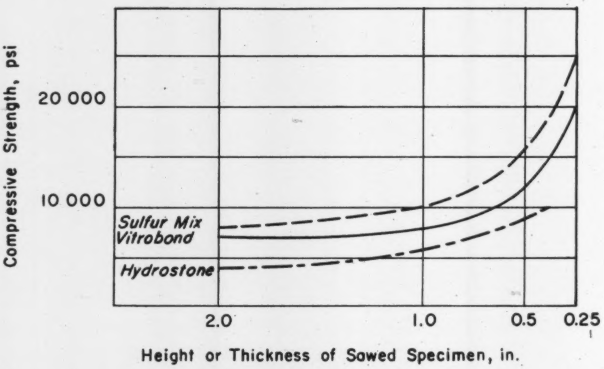

FIG. 8.—*L/D* Effect for Capping Materials.

Tests of sawed sections of 2 by 4-in. cylinders.

The accompanying Tables XI to XV cover five brief comparisons conducted during the period since February 1957 to reappraise and evaluate our capping procedure which is based on the laboratory prepared sulfur mix defined earlier. Some information on Vitrobond may be obtained by study of the paper by A. C.

Loewer et al (5) and their cited references. Vitrobond contains a plasticizer and cube or cylinder specimens of this capping material exhibit a bulging effect before failure; specimens of the sulfur mix containing granular material do not yield and fail suddenly by shattering.

PROPERTIES OF CAPPING MATERIALS—1951.

	Compressive Strength, 2 by 4-in. Cylinders, psi	Sonic Modulus of Elasticity, 2 by 2 by 10-in. bar, psi	
Vitrobond, 2 hr...	4960	(1 hr) 1.46	(18 hr) 1.84
Sulfur mix, 2 hr...	6720	(1 hr) 1.82	(18 hr) 1.90

TABLE XI.—SINGLE BATCH TEST.

¾-in. maximum size gravel aggregate concrete cured 28 days, compressive strength of 3 by 6-in. concrete cylinders, psi.

Top End Condition[a]	Ground Plane	Sulfur Mix Caps
Each specimen sawed to height, 5.75 in..........	6040 5210 5160 5810 5840	5870 5930 5300 5870 5950
Average..................	5610	5780

[a] Molded in plane-bottom steel molds.

TABLE XII.—SINGLE BATCH TEST.

¾ in. maximum size gravel aggregate concrete cured 5 days, compressive strength, psi.

Capping Method	Age of Cap	Steel Mold Top Capped	Paper Mold Two Caps	Molded by Operator
6 BY 12 IN. CONCRETE CYLINDERS				
Type III cement........	24 hr	3820	3820	I
Sulfur mix.....	2 hr	4010	4160	II
Vitrobond.....	2 hr	4030	3660	I
Hydrostone....	4 hr	4040	3960	II
Average.......	...	3975	3900	...
COMPANION 3 BY 6 IN. CYLINDERS—STEEL MOLDS				
Top sawed, ground plane.	...	4270, 4280		
Sulfur mix cap.	2 hr	4460, 4270		

Table XI is the only illustration showing *more than one* test specimen for each variable. In this single batch test comparison the intent was to make all specimens alike and the subgroups were selected by a system of randomizing. Despite this, a human element could have been involved since all specimens were not molded by one operator. Note that by deleting the second value from each column we could obtain respective averages of 5710 and 5750 which is an identity. However, it was preferred to report all the data for proper perspective.

In the succeeding illustrations no results could be excluded, even if a cap were faulty, since we molded only one specimen (afterwards taken at random) for each type cap. These examples are easy to examine and the comparison of different cappings may be made by inspection of each group.

Table XII contains the results of a demonstration test comparison arranged at short notice. The tests were also arranged to show the effect of using paper molds (requiring two caps) as well as identifying the operator who molded each specimen. Although representative of only one specimen for each variable, these data bear a remarkable resemblance to the averages for 60 specimens which were shown in the reproduction of Vidal's summary (1). Since the maximum size of gravel was $\frac{3}{4}$ in. it was possible to make smaller companion specimens in 3- by 6-in. molds and compare sulfur mix caps with ends ground to meet the planeness requirements of the specifications. These were supplemental data—four specimens molded by one operator.

In Table XIII results are shown for

3- by 6-in. cylinder specimens. The tests in all these illustrations are representative of specimens with smooth ends as made under laboratory and not field conditions, and all caps were applied by means of the vertical capping device. To determine relative strengths, the average of the 4 specimens on each line was utilized. When summarized, the average relative strengths for six levels in each column were: ground plane 96.0, sulfur mix 101.8, Vitrobond 99.3, and Hydrocal 102.8 per cent. Similar computation of average relative strengths, using averages of various groups of specimens of Boulder Canyon Project data (Table 64), yielded the same relative strengths as listed in Vidal's summary. (Note:—Data of individual specimens capped with plaster-cement seemed inappro-

TABLE XIII.—COMPARISON OF CAPPING MATERIALS.
¾ in. maximum size gravel aggregate concrete cured 7 days, compressive strength of 3 by 6-in. concrete cylinders, psi.

Capping Method	Ground Plane	Sulfur Mix	Vitrobond	Hydrocal	Average
Molded by Operator	A	B	A	B	
Plane Bottom Steel Molds—Top Capped					
Batch A	4490	4930	4680	(4910)[a, b]	4750
Batch B	5310	5390	5310	(5430)[a, b]	5360
Batch C	5610	5540	5680	5540	5590
Average, steel molds	5140	5290	5220	5290	....
Paraffined Paper Molds—Both Ends Capped					
Batch A	4330	4620	4350	4660[a]	4490
Batch B	4680	5060	4960	5430	5030
Batch C	5010	5490	5410	5430	5335
Average, paper molds	4670	5060	4910	5170	....

[a] Loose cap removed, replaced with sulfur mix cap.

[b] Numbers in parentheses indicate strength value not applicable to Hydrocal.

TABLE XIV.—SINGLE BATCH TESTS.
1½ in. maximum size limestone aggregate concrete at 2 ages, compressive strength of 6 by 12-in. concrete cylinders, psi.

	Age of Cap	Cured 7 Days		Cured 21 Days	
		Steel Mold Top Capped	Paper Mold Two Caps	Steel Mold Top Capped	Paper Mold Two Caps
Molded by Operator		I	II	I	III
Capping Method					
Sulfur mix	2 hr	4870	4420	6060	5230
	30 min	4620	....	5650	....
Vitrobond	2 hr	4400	4860	6070	5320
Hydrostone	4 hr	4480	4480	5800	5690
Hydrocal	4 hr	4600	4800	5820	5360[a]
Average	....	4590	4640	5940	5400

[a] Cap not thoroughly bonded to specimen.

priate and were excluded from the analysis.)

Average values for both steel and paper molds in Table XIII indicate that the three capping materials resulted in comparable strength values for a 5000 psi concrete. Since these data applied to 3 by 6-in. cylinders we were prompted to make a similar evaluation, by single batch tests, molding 6 by 12-in. cylinders under closely controlled laboratory conditions. However, the performance of well-paraffined paper molds was studied in each case.

Table XIV includes the results of a planned program comparing four capping materials on large cylinders made in two types of molds with strength levels governed by the curing time. By inspection it may be observed that similar values were obtained for each type of capping material—especially when it is realized that these are data of individual specimens. Relative strengths were determined for each specimen based on the average shown in each column. The average relative strengths for the various cappings were: sulfur mix 100, Vitrobond 100.5, Hydrostone 99.5, and Hydrocal 100 per cent. Thus, at the levels of this experiment, it appears that the 4 types of capping material are equal in performance. Interesting observations could be made about the suitability of the paper molds. Also, if capping both ends has an effect it can be noticed in these comparisons; this may have been a contributory factor in the tests of 21-day compressive strength. The "paper mold effect" would be more clearly defined if all specimens on each line were molded by the same operator.

Table XV is representative of everyday concrete produced commercially. All specimens were made in paraffined-paper molds and capped on both ends. The near-equality of results with each of four types of capping material is appar ent.

TABLE XV.—COMPARISON OF CAPPING MATERIALS.
1 in. maximum size gravel aggregate concrete at 2 ages,
compressive strength of 3 by 6-in. concrete cylinders—paper molds.

Capping Method		Sulfur Mix	Vitrobond	Hydrocal	Hydrostone	Average
Age of Cap		2 hr	2 hr	4 hr	4 hr	....
CURED 7 DAYS—BOTH ENDS CAPPED						
Mix 1	Molded by A	2100	2100	2040	2160	2100
Mix 2		2790	3140	2880	2680	2870
CURED 28 DAYS—BOTH ENDS CAPPED						
Mix 1	Molded by B	3650	3780	3610	3870	3730
Mix 2		4630	4660	4650	4680	4660
Average each type cap		3290	3420	3300	3350	....

Based on these tests, the reports of separate studies, and the reported observations of others, some concluding thoughts may be worthwhile:

1. The findings of the Bureau of Public Roads should be followed. These results are valued highly and should be utilized by Committee C-9.

2. Each laboratory should make check determinations of capping material at reasonable intervals to insure against inferior caps.

3. It appears that some comparisons

of vertical sulfur capping devices should be conducted to determine the effect of heating a recessed metal plate. There is a possibility that this factor could affect the planeness of cap.

4. Horizontal devices should be used with caution in capping high-strength concrete with sulfur mix.

5. The author's data show that greater attention should be given to planeness of cap, that planeness of capping plate does not necessarily insure that the cap is also plane.

6. We believe that any type cap should be securely bonded to the specimen.

7. The question arises, why was the statement permitting use of specimens with ground ends deleted from Methods C 31 and C 192 in 1949? The present requirements, in Methods C 31, Section 6(*a*) and in Methods C 192, Section 10(*a*), can be interpreted as permitting grinding of the ends of cylinders to meet planeness requirements (6), and

8. An optional procedure is needed for occasional testing of high-early-strength and steam-cured concrete promptly on arrival at the laboratory. It is possible that a moderate priced machine could be developed for grinding the ends of test cylinders to meet the planeness requirements.

MR. GEORGE WERNER (*author's closure*).—Mr. Helms' discussion adds much information to that furnished in the paper. His data indicate that cylinders with caps of sulfur cement or high-strength gypsum have, in many cases, strengths equal to or in excess of strengths of similar cylinders with hydraulic cement caps or with ground bearings. His discussion also calls attention to the common practice of using "home-made" sulfur cements instead of those that are sold commercially.

It appears that the use of sulfur cements for capping is increasing and therefore among Mr. Helms' concluding thoughts, No. 2 is appropriate. The use of sulfur mixtures that contain a plasticizer and which flow under load should be discouraged. Likewise, caps of home-made or commercial materials that warp, crack, or are not bonded to the concrete cylinder are unsuitable.

Mr. Helms' discussion of ground or lapped bearings on cylinders is timely. This method was not discussed in the author's paper and could be considered for testing cylinders in compression for research purposes because it eliminates any effect on strength that might be caused by a capping material.

The author's paper and the discussion leave some questions regarding the use of capping materials unanswered. It is hoped that their data will cause a sufficient interest to justify additional tests by other laboratories.

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SHEAR STRENGTH AND ELASTIC PROPERTIES OF SOIL-CEMENT MIXTURES UNDER TRIAXIAL LOADING*

By Glenn G. Balmer[1]

Synopsis

Triaxial shear results are presented in this paper from laboratory tests on cylinders molded from two granular and two fine-grain soils mixed with various amounts of portland cement at optimum moisture and maximum density as determined by ASTM Method D 558.[2] The cement content of these specimens was varied from zero to a percentage higher than that required to meet criteria for hardened soil-cement based on ASTM Wetting-and-Drying Test D 559[3] and Freezing-and-Thawing Test D 560.[4] Most of the specimens were cured 7, 28, and 90 days in a moist room at 73 F and tested triaxially in a moist condition.

Test procedures were developed to determine the shearing strength and the deformation of the specimens under triaxial load. The analyses of the test results showed that the coefficient of internal friction was relatively constant for cement-treated specimens of each soil regardless of cement content and age. Values averaged 0.96 for the granular and 0.73 for the fine-grain soil-cement mixtures. Cohesive strengths at age 28 days ranged from about 35 to 530 psi and depended upon cement content and type of soil.

The modulus of elasticity computed from the deformation measurements increased with cement content; average values at age 28 days varied from about 100,000 to 2,000,000 psi for the granular soil-cement mixtures and from about 260,000 to 760,000 psi for the fine-grain mixtures. Average values of Poisson's ratio seemed to be independent of cement content and age and varied between 0.05 and 0.20.

This paper covers one phase of an extensive laboratory investigation of the structural and physical properties of soil-cement mixtures. Previous publications (**1,2,3**)[5] have presented some of the results of experimentation on moisture-density, durability, compressive strength, flexural strength, and dynamic and static elastic properties. Other investigators (**4,5**) have also contributed to the development of this construction material by analyzing elastic behavior and triaxial strength. Studies are now in prog-

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* Presented at the Sixty-first Annual Meeting of the Society, June 22–27, 1958.

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[2] Methods of Test for Moisture-Density Relations of Soil-Cement Mixtures (D 558 – 57), 1958 Book of ASTM Standards, Part 4.

[3] Methods for Wetting-and-Drying Tests of Compacted Soil-Cement Mixtures (D 559 – 57), 1958 Book of ASTM Standards, Part 4.

[4] Methods for Freezing-and-Thawing Tests of Compacted Soil-Cement Mixtures (D 560 – 57), 1958 Book of ASTM Standards, Part 4.

[5] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 1203.

ress on volume change due to moisture and temperature changes and on the load-supporting characteristics of soil-cement pavements. An intimate knowledge of these physical and structural properties will improve construction practices and lead to rational design procedures.

An important phase of this study was the development of methods for making triaxial compression tests on specimens of soil-cement mixtures and for measuring axial and lateral deformations while the specimens were under load in the lateral pressure liquid. The loading data were used to determine the shearing strength of the materials by obtaining the cohesive strength and the coefficient of internal friction of each respective soil-cement mixture. This was accomplished by testing a series of companion specimens at lateral pressures of 0, 20, 40, and 60 psi. The lateral pressures were developed through a pneumatic load on kerosine, the lateral pressure medium. Data from the tests were analyzed to obtain the envelope to Mohr's circles which yielded the cohesion and coefficient of internal friction.

TABLE I.—GRADATION, LIQUID LIMIT, AND PLASTICITY INDEX OF SOILS.

	Per Cent by Weight			
	Soil No. 1, Sand	Soil No. 6, Sandy Loam	Soil No. 7, Silt Loam	Soil No. 8, Silty Clay Loam
Particles No. 4 to No. 10	27	20	0	1
Sand No. 10 to No. 40	37	22	1	2
Sand No. 40 to No. 200	32	39	7	6
Silt No. 200 to 0.005 mm	2	12	79	69
Clay minus 0.005 mm	2	7	13	22
Liquid limit	16	17	26	28
Plasticity index	not plastic	not plastic	7	9
AASHO[a] soil groups	A-1-b(0)	A-2-4(0)	A-4(8)	A-4(8)

[a] AASHO Designation M145-49.[6]

Axial and lateral deformation measurements were made on the specimens during loading by means of a compressometer utilizing unbonded wire gages devised especially for this purpose. These deformations were reduced to strains to compute the elastic properties of the mixtures.

The triaxial tests were made on soil-cement mixtures molded from four different soils typical of materials available for road and runway base construction. Cement contents used with these soils ranged from 0 to 16 per cent, and thus the data cover a wide range in shear values representing many cement-treated soils.

Both strength results and deformation characteristics as obtained from triaxial tests have been advocated as criteria in the design of flexible pavements (**6,7,8**). Data presented here will aid in determining whether these criteria apply to soil-cement pavements.

Materials and Test Specimens

The gradation, physical test constants, and AASHO Designation M145-49[6] which describe the four Illinois soils used in this study are shown in Table I. Soil No. 1 is a C-horizon pit-run sand with 4 per cent passing the No. 200 sieve, and soil No. 6 is a C-horizon light sandy loam with 19 per cent passing the

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[6] Standard Recommended Practice for the Classification of Soils and Soil-Aggregate Mixtures for Highway Construction Purposes (AASHO Designation M 145 - 49), *Highway Materials*, Part I: Specifications, 7th Edition, Am. Assn. State Highway Officials, p. 45 (1955).

No. 200 sieve. In contrast, soil No. 7 is a C-horizon silt loam, and soil No. 8 is a B-horizon silty clay loam. These latter two soils are similarly graded and contain over 90 per cent passing the No. 200 sieve; however, soil No. 8 contains organic material or foreign matter which hinders the cement reaction, and thus this soil does not respond to cement treatment as well as soil No. 7 which is free of deleterious material.

The following table lists the percentages of cement[7] mixed with each soil for this investigation. This cement was a blend of four brands of type I portland cement purchased in the Chicago market:

SOIL-CEMENT MIXTURES.

		Cement, per cent				
Soil No. 1	By weight.....	0	2	6	10	...
	By volume....	0	2.6	7.7	12.4	...
Soil No. 6	By weight.....	0	2	6	10	14
	By volume....	0	2.7	7.8	12.6	17.0
Soil No. 7	By weight.....	0	4	8	12	16
	By volume....	0	4.7	8.9	12.9	16.7
Soil No. 8	By weight.....	0	...	6	10	14
	By volume....	0	...	6.7	10.8	14.6

The ASTM Wetting-and-Drying[3] and Freezing-and-Thawing[4] tests show that 4 per cent of cement by weight is adequate to produce hardened soil-cement for the granular materials, soils Nos. 1 and 6; 8 per cent is adequate for the silt loam, soil No. 7; and 13 per cent is required for the silty clay loam, soil No. 8. Soil-cement mixtures containing less than these percentages are classified as cement-modified soils. Thus the program includes tests on specimens of untreated soil, cement-modified soil, and high-quality soil-cement. The terms "cement-treated soils" and "soil-cement mixtures" are sometimes used to include both cement-modified soil and soil-cement.

The optimum moisture content and the maximum density of each soil-cement mixture were determined in the laboratory by ASTM Method D558.[2] These values are listed in Table II under each soil for the cement contents tested. The materials were molded into 2.8 by 5.6-in. test cylinders, usually in batches of four, from constituents proportioned by weight from these predetermined moisture and density values. Duplicate specimens were molded a few days after the initial molding for testing as companion specimens. This provided two or more specimens at each lateral pressure and provided a check on molding and testing procedures.

The specimens were molded and cured in accordance with the Suggested Method of Making and Curing Soil-Cement Compression and Flexure Test Specimens in the Laboratory.[8] Predetermined weights of soil and cement were thoroughly mixed in a relatively dry state on a steel-topped table with a trowel. Next the computed quantity of water was added and thoroughly mixed to bring the mixture to the optimum moisture content. Sufficient weight of this mixture to provide a specimen of the design density 5.6 in. in length was placed in the 2.8-in diameter mold. The mold with a $\frac{1}{2}$-in spacer clip had previously been mounted on a piston base. The mixture was initially compacted by uniformly rodding the cross-section of the specimen to the point of refusal. Next, a separator disk was placed on the specimen, the spacer

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[7] The soil-cement mixtures were proportioned on a dry-weight basis. Later calculations were made to convert the cement contents to percentages by volume of loose cement to the compacted soil-cement mixture by the formula:

$$\text{Per cent cement by volume} = \frac{D - \dfrac{100D}{100 + P}}{0.94}$$

in which D is the oven-dry density of the mixture in lb per cu ft and P is the percentage of cement by weight. The assumption is made that 1 cu ft of loose cement (1 bag) weighs 94 lb.

[8] Procedures for Testing Soils, Am. Soc. Testing Mats. (ASTM Committee D-18), April 1958.

clip was removed, and the specimen was compacted to final density with dynamic loads acting on the pistons from both ends of the specimen.

After the specimens were molded, they were placed in the moist room and were removed from the molds on the following day. Moist curing of the cylinders was then continued until the end of 7, 28, or 90 days. The specimens, except those molded without cement, were capped just prior to testing with a mixture of portland cement, plaster, and water.

The program for soil No. 1, a granular soil, and soil No. 7, a fine-grain soil, included specimens cured by special procedures. The curing procedures were varied to explore the effects of different curing conditions and different moisture conditions on the elastic properties and the shearing strength of the mixtures. These specimens were first conventionally moist cured for 21 days. Certain of them were then placed in water at room temperature to soak for 7 additional days. Others were air dried for one day, then oven dried at 130 F for 3 days, and finally soaked for 3 days. Others were oven dried at 130 F for 6 days after one day of air drying. The special curing procedures were all completed at 28 days in order that the test results from these specimens could be compared with the 28-day moist-cured specimens.

TABLE II.—OPTIMUM MOISTURE CONTENT AND MAXIMUM DENSITY OF SOIL-CEMENT MIXTURES (ASTM METHOD D 558[2]).

Cement by Weight of Dry Soil, per cent	Soil No. 1		Soil No. 6		Soil No. 7		Soil No. 8	
	Maximum Density, lb per cu ft	Optimum Moisture Content, per cent[a]	Maximum Density, lb per cu ft	Optimum Moisture Content, per cent[a]	Maximum Density, lb per cu ft	Optimum Moisture Content, per cent[a]	Maximum Density, lb per cu ft	Optimum Moisture Content, per cent[a]
0	127	9.8	128	10.0	115	15.0	112	15.5
2	127	9.9	128	10.0	...	...	...	...
4	...	...	...	...	114	15.0	...	...
6	128	9.5	129	9.8	...	...	112	15.0
8	...	...	...	...	113	15.0	...	...
10	128	9.7	130	10.0	...	...	112	15.7
12	...	...	...	...	113	15.0	...	...
14	...	...	130	9.9	...	...	112	15.6
16	...	...	...	...	114	14.8	...	...

[a] By weight of soil-cement mixture.

TEST EQUIPMENT

The triaxial chamber used in these experiments, shown in Fig. 1 with a specimen ready for test, is similar to conventional soil testing equipment except that it has a higher axial load capacity. The circumferential pressure was produced by a pneumatic load on kerosine, the lateral pressure medium. Kerosine was used because it is a good insulator for electrical deformation measurements. Each specimen was encased in a thin Neoprene membrane before it was placed in the test chamber. A 90,000-lb hydraulic testing machine produced the axial load.

Since soil-cement mixtures have different physical properties than those of unmodified soils, the procedures for testing vary somewhat from conventional soil-testing procedures. The strains in cement-treated soils are much smaller for the same load than for untreated soil samples; consequently, it was necessary to measure deformations directly on the specimens rather than to measure volume changes and deformation from outside the test chamber.

The instrument (9) designed to make deformation measurements under tri-

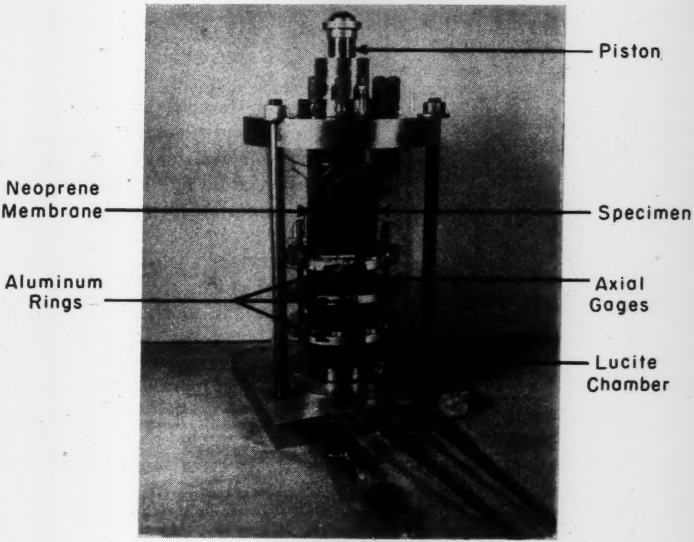
Piston
Neoprene Membrane
Specimen
Aluminum Rings
Axial Gages
Lucite Chamber

FIG. 1.—Triaxial Chamber with Encased Test Specimen.

axial loading is shown mounted on the specimen in Fig. 1. This compressometer consists essentially of three aluminum rings, two axial gages, two lateral gages, and two spacer bars, and it was mounted on the test specimens over the Neoprene membranes. The two axial unbonded elastic wire gages (see Fig. 2) encased in telescope tubing are attached to the upper ring and extend longitudinally along the specimens to the lower ring to measure axial deformation. The sensitive element of these gages consists of hard-tempered constantan wire 0.001 in. in diameter. Each elastic wire, which changes in electrical resistance as its length is changed, is fastened to a spring to increase its range sufficiently for the deformation encountered in these tests. An internal spring in each gage tends to extend the tubing and place the elastic

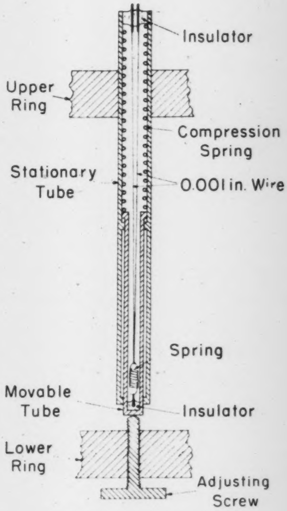
Insulator
Upper Ring
Compression Spring
Stationary Tube
0.001 in. Wire
Spring
Movable Tube
Insulator
Lower Ring
Adjusting Screw

FIG. 2.—Axial Gage.

wire in full tension. An internal stop limits this extension and protects the wire from breakage. Each gage is in contact with a screw in the lower ring which is used to adjust the tension in the wire before each test and to relax the wire when the instrument is not in use. Two similar gages are mounted in the middle ring (a split ring) to measure lateral deformation at the mid-height of the specimen.

The axial gages have a range of 0.06 in. in a 3-in. gage length and the lateral gages 0.03 in. in 2.8 in. gage length with a sensitivity of 10 units per million.

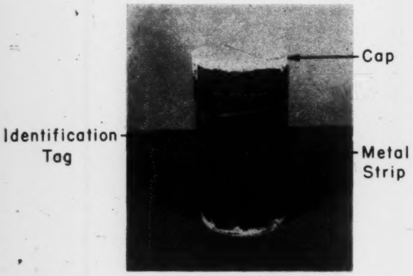

FIG. 3.—A Capped Specimen with Metal Strips for Measuring Deformation.

Spacer bars which hold the rings in position until they are secured on the test specimens are removed prior to testing. Each of the outside rings is attached by means of four pointed screws. The middle ring is mounted with two pointed screws. These bear through the Neoprene membrane against cylindrical metal strips previously attached to the test specimens with a quick-setting plaster as shown in Fig. 3. This provides a firm bearing surface which enables the instrument to detect small dilatations encountered early in the test. Compression springs about two alignment screws restrain the separation of the split ring and keep it properly aligned.

TEST PROCEDURE

The specimen was encased in a thin Neoprene membrane, and the compressometer was mounted on the specimen. The elastic wires of the axial gages were placed in full tension in order that the tension could be relieved as the specimen was compressed axially. Sufficient tension was removed from the lateral gages to allow for the anticipated increase in tension during dilatation.

Next, the specimen was placed in the triaxial chamber, and the membrane was clamped tightly to the pedestal and the loading head so that the kerosine would be sealed from access to the test cylinder.

After the chamber was closed and the electrical circuits were completed, the selected lateral pressure was applied to the specimen. This liquid pressure was held constant throughout the test as the axial load was gradually increased to the ultimate.

Deformation measurements were made at regular intervals of load without interruption. Generally the specimens were under load for about 3 min.

A minimum of eight specimens, two at each lateral pressure, were tested for each condition. The specimens were tested as soon as they were removed from the moist room except for those that were given special curing.

The compressometer was not used on soil specimens molded without cement. These specimens were not rigid enough to properly support the elasticity frame, and moreover, the deformations were too great for the range of the gages.

ANALYSIS OF RESULTS

The following tabulation provides a summary of notation:

τ, shearing strength, psi,
σ_n, normal stress, psi,

C, cohesive strength, psi,
tan ϕ, coefficient of internal friction,
ϕ, angle of internal friction,
σ_1, major principal stress, axial stress, psi,
σ_3, minor principal stress, lateral stress, psi,
a, unconfined compressive strength, psi,
b, slope of the principal stress equation,
E, static modulus of elasticity, psi,
μ, Poisson's ratio,
ϵ, strain,
D, density, lb per cu ft,
O, optimum moisture content, per cent by weight of compacted soil-cement mixture,

TABLE III.—TYPICAL TRIAXIAL STRENGTH RESULTS.
Soil No. 8, 6 per cent cement, moist-cured 90 days.

Lateral Stress, σ_3, psi	Axial Stress, σ_1, psi	Average Axial Stress, psi
0	435	
0	477	456
20	521	
20	532	526
40	600	
40	614	607
60	686	
60	705	696

P, percentage of cement by weight of dry soil, and
n, number of tests.

A typical set of triaxial strength data is given in Table III. σ_1 is the axial stress and σ_3 is the lateral stress which were acting on each cylinder at failure. Failure is defined as the maximum load supported by each specimen for the given applied lateral pressure.

These data may be shown graphically as in Fig. 4, where each point represents the result of a triaxial strength test. The line expresses a linear relationship between the principal stresses and illustrates how the strength increases with an increase of lateral pressure.

To determine the shearing strength, the forces on the test specimen at failure may be resolved into forces on the shearing plane in accordance with Mohr's method. In Fig. 5, a semicircle is drawn for the average axial stress for each lateral stress with these stresses as the extremities of the semicircle. The envelope to the semicircles is the relationship between the shearing strength and the stress normal to the shearing plane. The shearing strength is not a simple attribute of the material but is a function of the normal stress; whereas, the envelope is a function of the material.

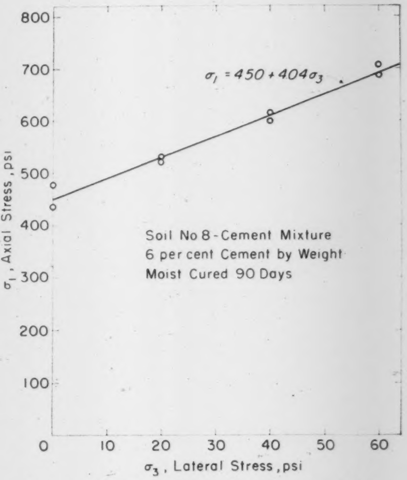

FIG. 4.—A Typical Principal Stress Relationship.

Rather than analyze each set of data graphically, the results presented in this paper were analyzed statistically. If we assume a linear equation for Mohr's envelope,

$$\tau = C + \sigma_n \tan \phi, \qquad (1)$$

the most probable values of the parameters *C* and tan ϕ can be determined by the method of least squares. The application of this method to the analysis of triaxial strength data is given in detail in a previous publication (10).

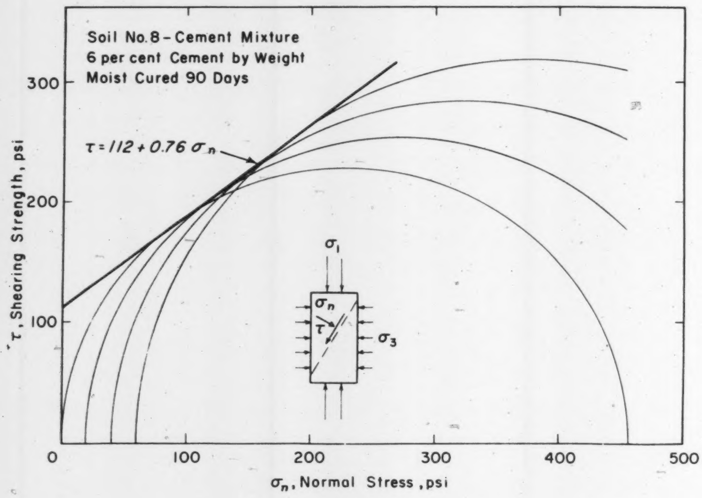

FIG. 5.—A Typical Mohr's Diagram.

TABLE IV.—TRIAXIAL TEST RESULTS OF SOIL NO. 6, SANDY LOAM.

Cement, per cent		Days Moist Cured	Shearing Strength		Number of Tests, n	Stress Relationship	
By Weight	By Volume		C,[a] psi	$\tan \phi$[a]		a,[a] psi	b[a]
0	0	1	5	0.79	11	19	4.27
2	2.7	7	35	0.94	13	160	5.31
2	2.7	28	35	1.05	11	174	6.25
2	2.7	90	56	1.02	10	273	6.02
6	7.8	7	84	1.10	8	435	6.69
6	7.8	28	150	1.04	8	749	6.20
6	7.8	90	203	0.99	11	975	5.79
10	12.6	7	250	0.88	10	1107	4.89
10	12.6	28	317	0.98	15	1511	5.69
10	12.6	90	421	1.01	8	2044	5.90
14	17.0	7	376	0.98	9	1786	5.65
14	17.0	28	531	0.91	13	2412	5.15
14	17.0	90	547	1.05	8	2730	6.23
Arithmetic mean[b]				1.00 (ϕ = 45 deg)			5.81
Standard deviation[b]				0.06			0.49

[a] Least squares (**10**) values of the parameters of the equation, $\tau = C + \sigma_n \tan \phi$, and the corresponding principal stress equation, $\sigma_1 = a + b\sigma_3$.

[b] 0 per cent cement content not included.

TABLE V.—TRIAXIAL TEST RESULTS OF SOIL NO. 1, SAND.

Cement, per cent		Days Moist Cured	Shearing Strength		Number of Tests, n	Stress Relationship	
By Weight	By Volume		C, psi	tan ϕ		a, psi	b
0	0	1	5	0.79	9	22	4.26
2	2.6	7	34	0.89	8	152	4.96
2	2.6	28	41	0.99	8	196	5.77
2	2.6	[a]	35	1.02	8	170	5.99
2	2.6	[b]	29	1.01	9	143	5.93
2	2.6	[c]	63	1.24	8	358	8.06
2	2.6	90	56	0.97	8	264	5.56
6	7.7	7	125	0.94	9	579	5.33
6	7.7	28	202	0.84	8	867	4.60
6	7.7	[a]	171	0.95	8	796	5.41
6	7.7	[b]	176	0.90	9	793	5.07
6	7.7	[c]	359	1.07	11	1833	6.45
6	7.7	90	236	0.94	9	1091	5.32
10	12.4	7	269	0.85	9	1160	4.65
10	12.4	28	359	0.84	8	1539	4.60
10	12.4	90	401	0.92	10	1827	5.18
Arithmetic mean[d]				0.91 (ϕ = 42 deg)			5.11
Standard deviation[d]				0.05			0.41

[a] 21 days moist cured plus 7 days soaked.

[b] 21 days moist cured plus one day air dried plus 3 days oven dried at 130 F plus 3 days soaked.

[c] 21 days moist cured plus one day air dried plus 6 days oven dried.

[d] Special cured and 0 per cent cement content specimens not included.

TABLE VI.—TRIAXIAL TEST RESULTS OF SOIL NO. 7, SILT LOAM.

Cement, per cent		Days Moist Cured	Shearing Strength		Number of Tests, n	Stress Relationship	
By Weight	By Volume		C, psi	tan ϕ		a, psi	b
0	0	1	8	0.52	11	28	2.71
4	4.7	7	75	0.60	9	267	3.14
4	4.7	28	95	0.66	8	352	3.45
4	4.7	[a]	84	0.63	8	303	3.27
4	4.7	[b]	55	0.51	8	179	2.68
4	4.7	[c]	102	1.15	9	545	7.13
4	4.7	90	112	0.76	8	453	4.09
8	8.9	7	97	0.76	8	391	4.05
8	8.9	28	152	0.67	8	570	3.53
8	8.9	[a]	152	0.61	9	540	3.17
8	8.9	[b]	85	0.94	8	394	5.31
8	8.9	[c]	200	1.29	8	1172	8.55
8	8.9	90	226	0.76	10	909	4.06
12	12.9	7	171	0.63	9	620	3.27
12	12.9	28	177	0.89	8	785	4.93
12	12.9	90	303	0.76	9	1225	4.07
16	16.7	7	224	0.72	8	871	3.79
16	16.7	28	227	0.91	14	1023	5.08
16	16.7	90	391	0.79	8	1612	4.25
Arithmetic mean[d]				0.74 (ϕ = 37 deg)			3.98
Standard deviation[d]				0.09			0.57

[a] 21 days moist cured plus 7 days soaked.

[b] 21 days moist cured plus one day air dried plus 3 days oven dried at 130 F plus 3 days soaked.

[c] 21 days moist cured plus one day air dried plus 6 days oven dried.

[d] Special cured and 0 per cent cement content specimens not included.

The computed results for the strength data from the experiments are listed in Tables IV, V, VI, and VII.

TABLE VII.—TRIAXIAL TEST RESULTS OF SOIL NO. 8, SILTY CLAY LOAM.

Cement per cent,		Days Moist Cured	Shearing Strength		Number of Tests, n	Stress Relationship	
By Weight	By Volume		C, psi	tan ϕ		a, psi	b
0	0	1	13	0.43	11	41	2.30
6	6.7	7	90	0.69	9	342	3.60
6	6.7	28	97	0.80	8	405	4.33
6	6.7	90	112	0.76	8	450	4.04
10	10.8	7	138	0.70	9	530	3.69
10	10.8	28	180	0.69	12	685	3.62
10	10.8	90	234	0.71	8	908	3.75
14	14.6	7	185	0.60	8	655	3.14
14	14.6	28	198	0.74	8	784	3.92
14	14.6	90	274	0.77	8	1110	4.12
Arithmetic mean[a]				0.72 (ϕ = 36 deg)			3.80
Standard deviation[a]				0.06			0.33

[a] 0 per cent cement content not included.

Equation 1 is mathematically related to the equation between the principal stresses,

$$\sigma_1 = a + b\sigma_3 \text{, ...(2)}$$

in which a is the unconfined compressive strength and b is the slope of the straight line or the rate of change of the axial stress with respect to the lateral stress.

The transformation

$$\tau = \frac{\sigma_1 - \sigma_3}{2} \cos \phi \text{. . . . .(3)}$$

and

$$\sigma_n = \frac{\sigma_1 + \sigma_3}{2} - \frac{\sigma_1 - \sigma_3}{2} \sin \phi \text{. . . .(4)}$$

can be applied to Eq 1 to obtain

$$\sigma_1 = \frac{2C \cos \phi}{1 - \sin \phi} + \frac{1 + \sin \phi}{1 - \sin \phi} \sigma_3 \text{. . . .(5)}$$

which is equivalent to Eq 2. This equation defines an average axial strength of the material for each lateral stress.

Individual stress conditions can be computed from the above equations after the characteristics of the materials have been established. Caution should be exercised in extrapolating the values beyond the range of the experimental data because the envelope to Mohr's diagram may be curved. If this is true, the value of ϕ will probably be smaller at higher stresses.

Typical stress-strain curves are shown in Fig. 6, in which the abscissa is strain

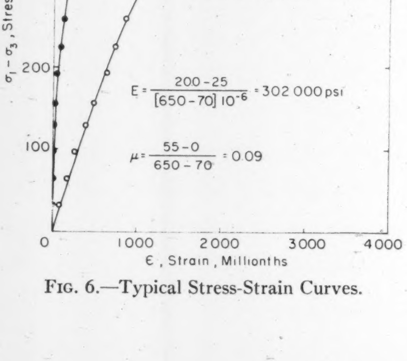

FIG. 6.—Typical Stress-Strain Curves.

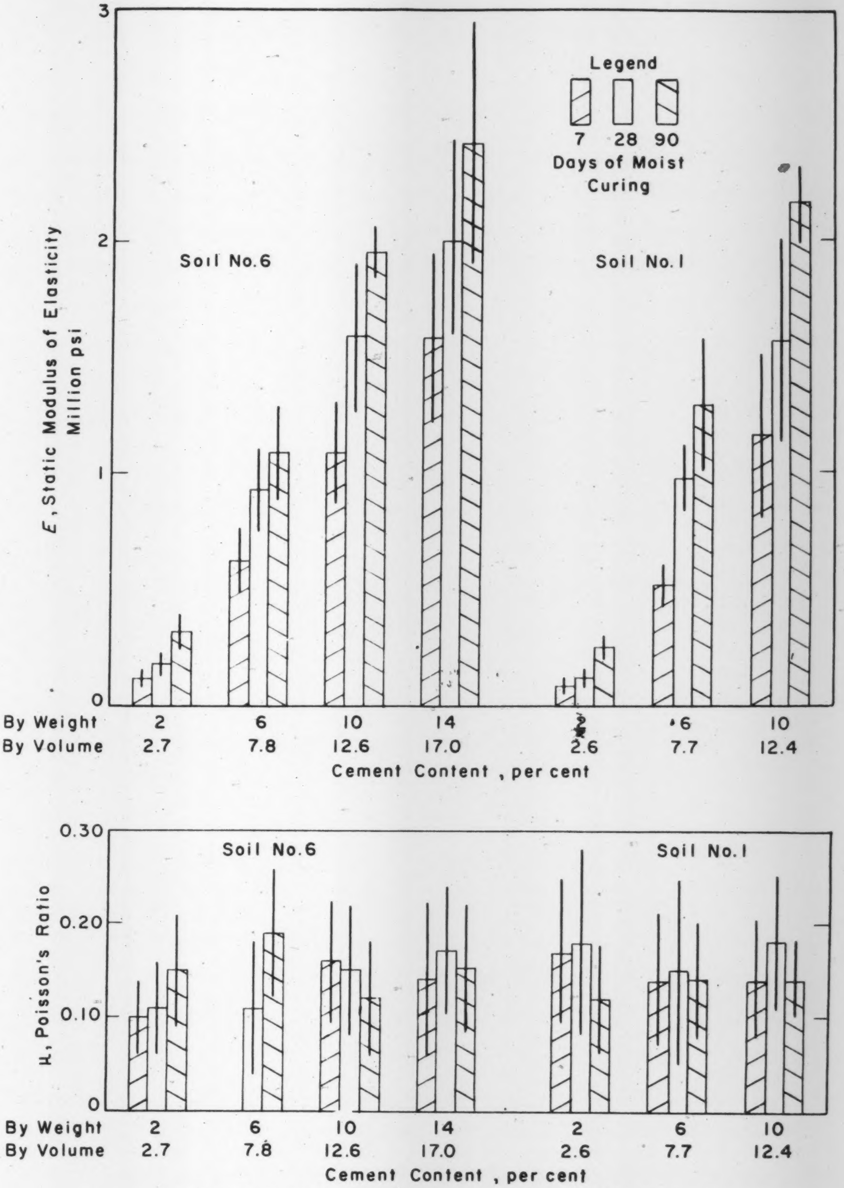

FIG. 7.—Elastic Properties of Granular Soil-Cement Mixtures.

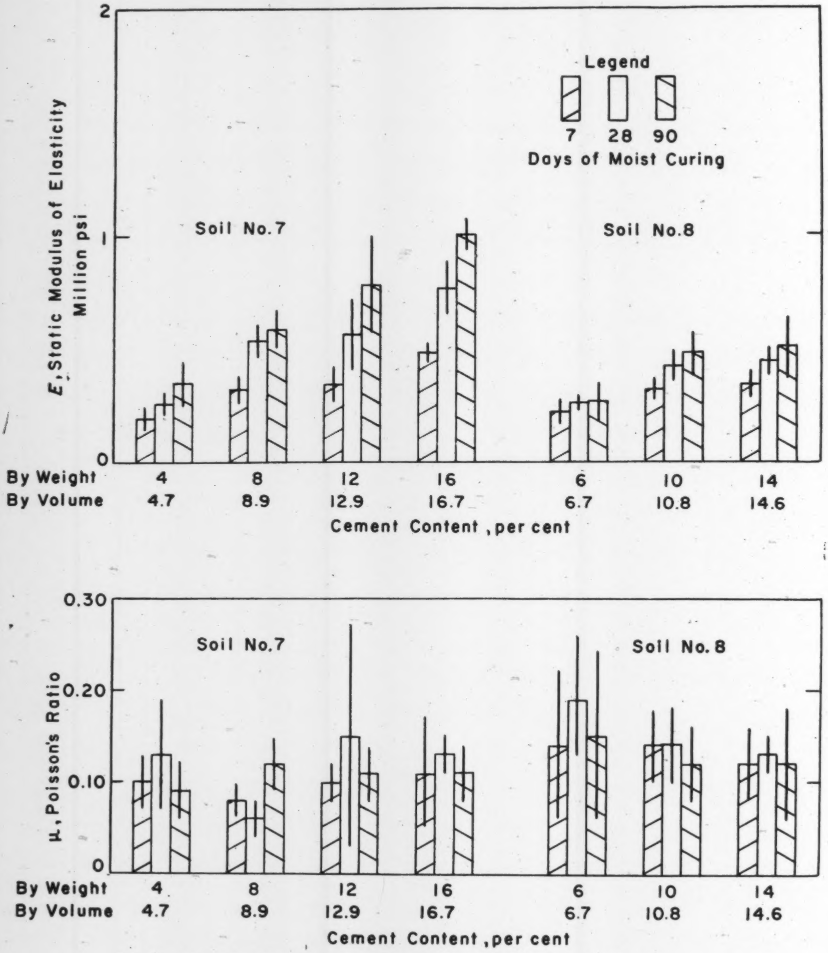

FIG. 8.—Elastic Properties of Fine-Grain Soil-Cement Mixtures.

and the ordinate is the stress difference. Typical calculations of E, the static modulus of elasticity in compression, and μ, Poisson's ratio, are shown on the graph.

Note →

E was computed as a secant modulus from the ascending phase of the first cycle of loading between about 10 and 40 per cent of the ultimate strength of the specimen, presumably within the elastic range of the material. Poisson's ratio was computed as the ratio of lateral strain to axial strain over the same stress range.

A previous paper (3) has shown that the values of the static modulus in compression for soil-cement mixtures are

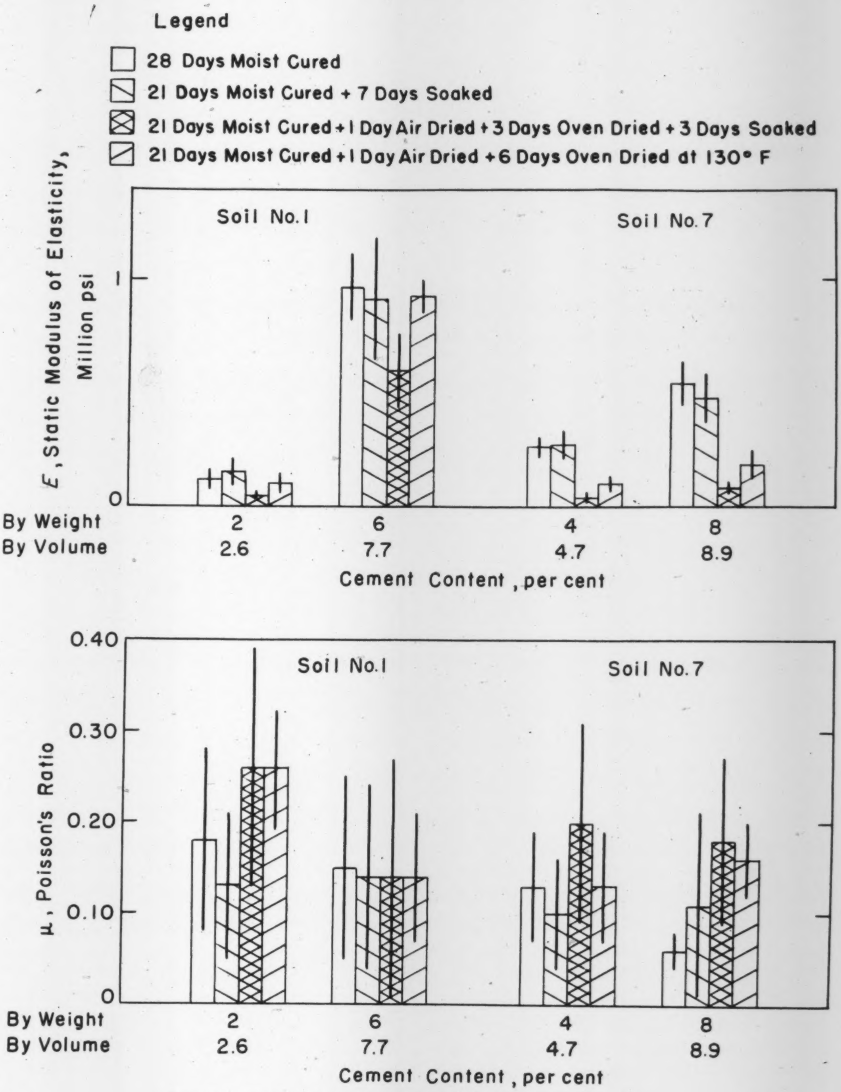

FIG. 9.—Elastic Properties of Specially Cured Specimens.

about 60 to 75 per cent of the dynamic modulus and also that the dynamic modulus and the static modulus in flexure are about equal.

The effect of the different lateral pressures on the elastic properties of the soil-cement mixtures was not significant over the small range of lateral pressures, 0 to 60 psi, used in these tests. Consequently, the values for the modulus of elasticity and Poisson's ratio were averaged over all lateral pressures. The average results are represented by the bar graphs in Figs. 7, 8, and 9. A vertical line has been drawn through each bar graph equal in length to a spread of one standard deviation on each side of the mean; this provides a measure of the dispersion of the data for each cement content and age.

DISCUSSION OF RESULTS

The strength data tabulated in Tables IV through VII show that the cohesive strength, *C*, and the unconfined compressive strength, *a*, increased with cement content and age.

The coefficient of internal friction, tan ϕ, was relatively constant for each soil. Its value was unrelated to the percentage of cement mixed with the soil. Consequently, the values of tan ϕ have been averaged and the standard deviation computed for the test results on each soil, excluding the specially cured specimens. Similar remarks can be made in regard to *b*, the slope of the line relating the principal stresses. These computed values are shown at the bottom of their respective columns in each table.

The coefficient of internal friction was larger for the cement-treated granular soils than for the cement-treated fine-grain soils as revealed by the average values of tan ϕ of 0.91 and 1.00 for soils Nos. 1 and 6 in contrast to 0.74 and 0.72 for soils Nos. 7 and 8. The coefficient of internal friction was also larger for the cement-treated soils than for the untreated soils. The above values for the soil-cement mixtures may be compared with 0.79 for the granular untreated soils and with 0.52 and 0.43 for untreated soils Nos. 7 and 8.

Granular soils have little or no cohesion, but the cohesive strength of soil-cement mixtures of these materials increased rapidly as the cement content was increased. The cohesive strength of the granular soil-cement specimens exceeded that of the fine-grain soils for the same cement content at 6 per cent and higher with one exception, as shown by Tables IV through VII.

Drying cement-treated specimens markedly increased their coefficient of internal friction, as shown by values of 1.24 and 1.07, for soil No. 1 (Table V) and values of 1.15 and 1.29 for soil No. 7 (Table VI). In addition, drying without resoaking increased the cohesive strength greatly.

The cohesion was not as great for the specimens tested in a soaked condition as for specimens tested in a moist condition. This was pronounced for specimens which had been dried prior to soaking (Tables V and VI). When specimens are dried, the water films around the particles become very thin and exert high surface tension forces. This causes the specimens tested in a dry state to be stronger than those tested in a moist state. The soaked specimens adsorb water, and thus the surface tension forces are reduced and the strengths are reduced.

While the range in lateral pressures between 0 and 60 psi applied in these tests is small compared to the pressures applied for concrete and rock (10,11), it is a realistic range for road or runway base materials. The stresses developed from traffic on soil-cement pavements probably are within the range of stresses covered by these experimental data.

Failure patterns for test specimens of

this study can be visualized from Figs. 10 and 11. In some cases a definite shear plane was observed; in other cases the specimens cracked or crumbled so that no failure pattern was observed; while in still other cases the specimens bulged as in Fig. 11. This latter type of failure was prevalent for the specimens with no cement or low cement content.

The modulus of elasticity in compression for specimens of cement-treated mixtures of granular soil No. 6 increased from 120,000 psi with 2 per cent cement to 1,600,000 psi with 14 per cent cement after 7 days moist curing and to 2,400,000 psi after 90 days moist curing. The increase in modulus for granular soil No. 1 was comparable.

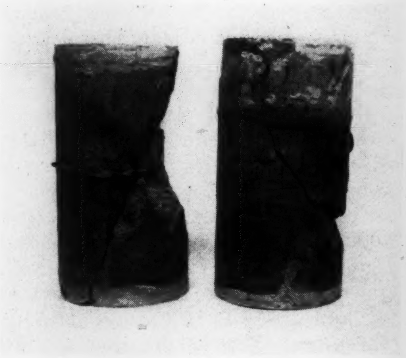

FIG. 10.—Specimens After Failure.

The modulus for fine-grain soil No. 7 increased from 200,000 psi with 4 per cent cement to 500,000 psi with 16 per cent cement after 7 days moist curing and to 1,000,000 psi after 90 days moist curing. The modulus for fine-grain soil No. 8 increased from 220,000 psi for 6 per cent cement cured 7 days to 500,000 psi for 14 per cent cement cured 90 days.

Most of this discussion has referred to average results; however, the scatter of the individual tests can be judged from the standard deviation of the data illustrated by the vertical lines through the bar graphs of Figs. 7, 8, and 9. Theoretically two thirds of the test data are included between the extremities of these lines.

The effect of the different curing procedures and test conditions on the elastic properties of the soil-cement specimens can be observed in the graphs of Fig. 9. Drying the cylinders decreased the modulus of elasticity slightly for the sandy soil No. 1 and decreased the modulus 60 per cent for the silty soil No. 7. Even though all of the specially cured specimens were moist cured 21 days, drying them and resoaking them after the initial curing decreased the modulus materially. The decrease was between 35 and 60 per cent for soil No. 1 and over 80 per cent for soil No. 7 when compared with the 28-day moist cured specimens.

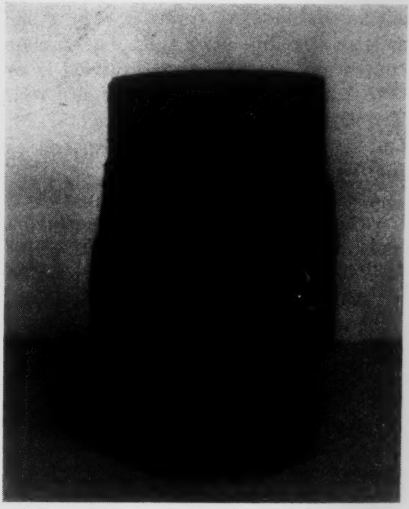

FIG. 11.—Failure of Low Cement Content Specimen.

The cement content or age had little influence on Poisson's ratio for any of the cement-treated soils. The average values for the moist-cured specimens varied between 0.10 and 0.20 for the granular soils and between 0.05 and 0.20 for the fine-grain soils. The composite average was slightly higher for the sandy soils than for the silty soils.

SUMMARY

The shearing strength and elastic properties of soil-cement mixtures have been determined by laboratory tests on specimens molded from two granular soils and two fine-grain soils. The cement contents of these specimens varied from 0 to 16 per cent by weight of dry soil. The cylinders were moist-cured 7, 28, and 90 days except for a few specimens that were given special curing.

The triaxial shear results showed that the cohesive strength for specimens moist cured 28 days increased from 35 psi for 2 per cent cement to 531 psi for 14 per cent cement for the sandy loam-cement mixtures and from 95 psi for 4 per cent cement to 227 psi for 16 per cent cement for the silt loam-cement mixtures. Thus the rate of increase in strength with cement content was greater for the granular soil-cement mixtures than for the silty soil-cement mixtures.

The cohesive strength also increased with age. In general the 90-day values increased as much as or more than 50 per cent of the 7-day values.

The specimens tested in a dried condition manifested a significant increase in cohesion for the silty soil-cement mixtures and a more than 50 per cent increase for the granular soil-cement mixtures as compared with the 28-day moist-cured specimens. Those specimens tested in a soaked condition showed a lower cohesion.

The coefficient of internal friction of conventionally cured specimens was independent of the cement content and age but varied with the type of soil with average values of 0.91 (ϕ = 42 deg) and 1.00 (ϕ = 45 deg) for the granular soil-cement mixtures and of 0.74 (ϕ = 37 deg) and 0.72 (ϕ = 36 deg) for the fine-grain soil-cement mixtures tested. These average values were larger for the specimens molded with cement than for the specimens molded from untreated soil as indicated by the values 0.79 (ϕ = 38 deg) for the granular soil specimens and 0.52 (ϕ = 27 deg) and 0.43 (ϕ = 23 deg) for the fine-grain soil specimens.

Specimens tested dry showed values of tan ϕ of 1.24 (ϕ = 51 deg) and 1.07 (ϕ = 47 deg) for the granular soil-cement mixtures and of 1.15 (ϕ = 49 deg) and 1.29 (ϕ = 52 deg) for the silty soil-cement mixtures. These values are considerably greater than those for specimens conventionally moist-cured.

The modulus of elasticity increased with cement content. The sandy loam soil-cement mixtures showed average 28-day values of E = 200,000 psi for 2 per cent cement as compared with E = 2,000,000 psi for 14 per cent cement. E also increased for the silt loam soil-cement mixtures from 260,000 psi at 4 per cent cement to 760,000 psi at 16 per cent cement. The modulus also increased with age. In some cases the 90-day values were double the 7-day values, but the increase averaged about 50 per cent of the 7-day values.

Although drying the specimens increased the strength, it decreased the modulus of elasticity. The decrease was slight for the granular soil-cement mixtures but was as much as 60 per cent for the silty soil-cement mixtures.

Poisson's ratio exhibited a random variation with cement content and age and averaged 0.14 for the granular soil-cement mixtures and 0.12 for the fine-grain soil-cement mixtures.

The conclusions drawn in this paper refer to the soil-cement mixtures tested. It is not intended that these results should eliminate the necessity of laboratory tests for individual soils. On the contrary, the triaxial test method provides procedures and an additional tool for evaluating the physical characteristics of such materials.

Acknowledgment:

Credit is due Gervaise E. Monfore for the novel design and expert performance of the unbonded wire gages and Melvin S. Abrams for helpful suggestions in developing testing procedures. This experimental investigation was under the direction and guidance of Earl J. Felt, Manager, Transportation Development Section, Portland Cement Assn., Chicago, Ill.

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DISCUSSION

MR. HAROLD J. GIBBS[1] (*by letter*).—The author has presented excellent data to demonstrate the effects of cement content and curing on the shearing strength of soil-cement. It is well presented and the writer is impressed with the systematic program of tests made.

As the paper shows, it is quite apparent that the principal effect is the change in cohesion with significant increases of several times in value as cement content and curing time increases. On the other hand, there were small changes in tan ϕ but still quite significant differences in the average values of tan ϕ for the different soils. Since most other papers on strength of soil-cement deal with results of only the unconfined compression test which implies cohesion, and this paper is a unique gathering of triaxial shearing test results, the writer believes that more significance can be obtained from data on the values of tan ϕ.

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[1] Bureau of Reclamation, U. S. Department of the Interior, Denver, Colo.

In order to thoroughly understand the characteristics of tan ϕ, the type of test should be described in respect to whether it was (1) the drained test, (2) the consolidated undrained test, or (3) undrained test. It is understood by the writer that the values of tan ϕ and cohesion are those determined from applied stresses, but it is not understood whether drainage was permitted through porous end plates or whether the specimens were fully sealed during the tests. It was mentioned, however, that the test was conducted in a rather short time of about 3 min and soil-cement specimens were capped. These viewpoints would be quite important in making the values of tan ϕ more meaningful. Possibly they would also affect the cohesion characteristics some. However, the cohesion values have large differences and the paper very effectively shows the trends in cohesion for the varying soil-cement mixtures.

Assuming that all tests were equivalent to the undrained shear test since the soil-cement specimens were capped and drainage is not mentioned, the following trends in tan ϕ should logically exist:

1. Coarser soil should naturally show higher tan ϕ values than finer soils because lower pore pressures should be expected in coarser soils which usually consolidate less under load and contain lower moisture contents.

2. Cement content also affected tan ϕ because pore pressures were probably lower, and it was explained that cement appreciably reduced the consolidation, requiring considerable refinements to measure the volume change.

3. It was also noted that specimens which were oven dried before testing showed appreciably greater values of tan ϕ than similar specimens cured normally and soaked. This could partially be attributed to lower pore pressures resulting from low moisture content and small volume change as well as higher general strength of these specimens.

4. Finally when tan ϕ in terms of effective pressures (that is, applied pressures minus pore pressures) is considered, fine-grain soils generally show lower values of tan ϕ than coarser soils. These soils may have this trend but in addition, the above items probably have some effect on the results and should be given consideration.

This paper is of particular significance over other papers concerning the strength of soil-cement in that it has presented shear characteristics in terms of complete triaxial shear tests rather than only the unconfined compressive strength. This gives added data on the relationship of strength and pressure. The purpose of this discussion is to suggest additional interpretations which could result when full consideration is given to the conditions of the test.

MR. G. G. BALMER (*author's closure*).—The triaxial results in the paper were computed from applied stresses on undrained specimens; however, the specimens were in a moist rather than in a saturated condition when tested. Cement-treated specimens are much stiffer than untreated soils; therefore, less consolidation took place for these specimens during the triaxial tests than for untreated soil specimens. Pore pressures were not measured during these tests.

The remarks of the discussion substantiate the conclusions of the author and help to explain the reasons for the results.

SOIL MOISTURE SUCTION—ITS IMPORTANCE AND MEASUREMENT*†

By E. Penner[1]

Synopsis

Perhaps the greatest development in soil mechanics has been in understanding the mechanical behavior of saturated soils. More recently attempts have been made to deal with soils in the unsaturated state. Under these conditions the behavior of the soil depends to a large extent on the moisture status, which for many purposes can be usefully expressed in terms of "suction," commonly known also as moisture tension or negative hydraulic potential.

The identity of suction with relative vapor pressure and hence free energy is given together with the advantages of measuring the former in the region of high relative vapor pressures. Since the behavior of water in soils and many related porous materials is similar, the suction concept is not limited to soil.

Both the apparatus and technique used for determining soil moisture suction and unsaturated permeability are described together with the use of the methods in soil engineering.

Many of the practical problems that arise with the use of materials of a porous nature result from their interaction with water. In addition to the influence of water on the performance of porous materials, there is the related aspect of understanding the properties of held water, in particular as it affects moisture transmission.

The problems that result from the interaction with water vary with the type or porous material and the service expected from it. For example, the problems may range from those concerned with moisture transmission in building walls to those involved in the determination of shear strength in soils. Acknowledging these differences, there is a distinct need for a more adequate way of describing the moisture status in porous materials than by water content on a weight percentage or per cent saturation basis. Such a measure should describe the held moisture in terms of its tendency to move between similar as well as dissimilar materials. Held moisture in terms of moisture content often gives no real indication of the tendency of the moisture to move because two different materials may be in equilibrium with each other at widely different moisture contents. Also similar materials with a different moisture history may not be in moisture equilibrium at the same water content. This second phenomenon involves hysteresis and will be referred to later.

The concept of suction that has been widely used in the field of soil science for many years (1)[2] provides a basis for a rational and useful method of describing the moisture status of porous materials. More recently it has been usefully employed to a limited extent in some aspects of soil mechanics and in other engineering fields concerned with the behavior of moisture in other porous materials. Since the suction concept has been shown to be useful and has led to a better understanding of the behavior of moisture in porous material, it deserves much wider attention. This paper is concerned with a simple description of the nature of suction, some methods of measuring it, and finally it gives a brief account of its application to some soil engineering problems.

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* Presented at the Sixty-first Annual Meeting of the Society, June 22–27, 1958.

† This is a contribution from the Division of Building Research, National Research Council, Canada, and is published with the approval of the Director of the Division.

[1] National Research Council, Division of Building Research, Ottawa, Canada.

MOISTURE SORPTION BY POROUS MATERIALS

Soils belong to a class of adsorbents which give a typically S-shaped relationship when the moisture content is plotted as a function of the equilibrium relative vapor pressure. These curves are commonly referred to as sorption isotherms. Several such curves are shown in Fig. 1, including some for several materials other than soils.

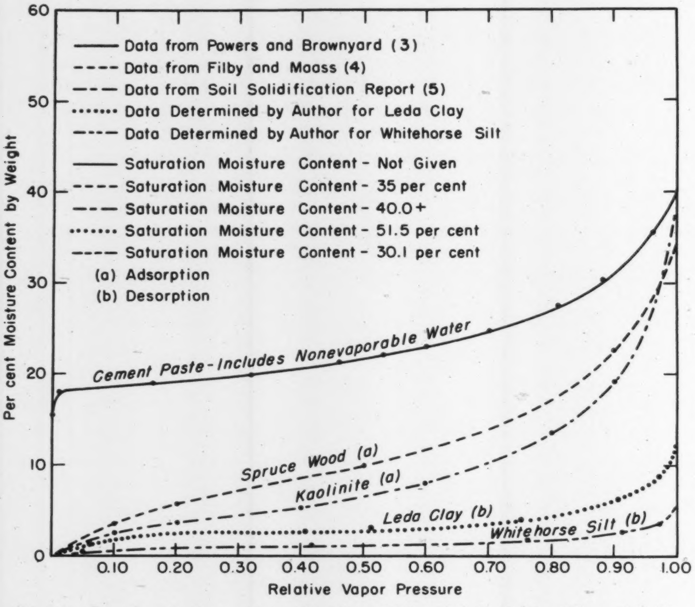

FIG. 1.—Sorption Isotherms for a Number of Materials Including Natural Soils.

Although a detailed discussion of the various mechanisms of moisture sorption is beyond the scope of this paper, a brief reference to the subject is useful in describing the suction concept.

Moisture suction in partially saturated materials (of the class referred to above) at low moisture content arises from the attraction between water molecules and the solid. This type of attraction is characterized by physical forces rather than chemical and is normally considered to be reversible. In the case of clays, a part of the sorption arises from the hydration of the exchangeable ion, but as can be seen from Fig. 1 the general shape of the sorption curve is much the same.

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[2] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 1216.

The physical arrangement of water molecules in porous materials is not well understood, but it is commonly assumed that the molecules are spread over the solid surface at low relative vapor pressures. With increasing vapor pressure, the thickness of the sorbed layer increases until at high vapor pressures the increase in held moisture results in the filling of pores. In this range the forces of sorption can be considered as arising from the surface tension forces operating in the concave water menisci in partially filled pores. In the intermediate vapor pressure range both kinds of sorption are probably operative. The forces of attraction acting on the water molecules are reflected in a reduction in vapor pressure, relative to the vapor pressure over a flat water surface at the same temperature. Relative vapor pressure is therefore a direct measure of the forces operating at the surface of the held water.

The relationship between relative vapor pressure and the radius of curvature of the water meniscus is given by the Kelvin equation:

$$\ln p/p_0 = -\frac{2\,\gamma\,M}{r\,d\,RT} \quad \ldots\ldots\ldots (1)$$

where:

p = the vapor pressure over the curved surface,
p_0 = the vapor pressure of free water,
γ = the surface tension of water,
M = the molecular weight of water,
d = the density of the water,
R = the gas constant,
T = the absolute temperature, and
r = the radius of curvature of the meniscus.

This equation shows that the equilibrium vapor pressure, p, over a curved water meniscus is reduced below the vapor pressure p_0 as a result of surface tension. As will be shown later, relative vapor presssure, though a rational measure of the forces with which water is held, is not always a convenient one, particularly at high vapor pressures when many pores are filled and other larger pores contain menisci. Under these conditions, it is often more convenient and useful to express the forces in another way that is directly related to the negative hydraulic head, or "suction" of the liquid phase.

TABLE I.—THE RELATIONSHIPS BETWEEN RELATIVE VAPOR PRESSURE, RADIUS OF CURVATURE OF MENISCUS, EQUIVALENT HEIGHT OF COLUMN OF WATER *h*, AND pF.

p/p_0, per cent	Radius of Curvature of Meniscus, r	Equivalent Height of Water, h, cm	h, psi	Schofield's pF
0.08...	0.000147	10^7	1.42×10^5	7
49.0....	0.00147	10^6	1.42×10^4	6
93.0....	0.0147	10^5	1.42×10^3	5
99.3....	0.147	10^4	1.42×10^2	4
99.9....	1.47	10^3	1.42×10	3
99.99...	14.7	10	1.42	2
99.999..	147.0	10	1.42×10^{-1}	1
100.0....	00.0	0	0	

The concept of suction is based upon the height, h, to which water rises in an ideal capillary tube. This height, h, is dependent on r, the radius of the tube. If the contact angle between the capillary tube and the liquid is zero, the radius of curvature of the meniscus and the radius of the capillary tube will be equal. The height of the capillary rise, h, being related to the curvature of the water surface, can therefore be used as a measure of the potential of the water at the surface of the meniscus having the same radius, *r*, even though a complete water column does not exist. This is the measure used to express suction. For convenience, Schofield (6) has chosen to express the height of rise in terms of log (− *h*), which is now commonly known as pF. Relative vapor pressure, being related to the curvature of the meniscus, is also related to the suction, *h*. The relationship, which is well known, may be derived from the Kelvin equation.

From the height-of-capillary-rise equation:

$$gh = \frac{2\gamma}{dr} \quad \text{.............(2)}$$

where:

g = gravitational constant,
h = height-of-capillary rise,
γ = surface tension of water,
d = density of the water, and
r = radius of the capillary.

Substitution of *gh* in the Kelvin equation leads to the relationship:

$$h = -\frac{RT}{Mg} \ln p/p_0 \quad \text{.........(3)}$$

Regarding the use of free energy as a measure of the moisture potential, it may be pointed out that the use of the suction concept for a salt-free system does not lose identity with the free energy concept. Although *h* measured directly will not include the effect of salt, vapor pressure does. When the held water has reached a state of equilibrium with the vapor in the surrounding environment, the free energy of the vapor and the sorbed moisture will be equal. Consequently, the free energy of the vapor may be used to calculate the free energy of the sorbed phase from the expression:

$$\Delta F = \frac{RT}{M} \ln p/p_0 \quad \text{..........(4)}$$

It is of interest to note the similarity between Eqs 3 and 4, as pointed out by Schofield, and its relationship to the quantity p/p_0, the relative vapor pressure. The relationship between relative vapor pressure, radius of curvature of the meniscus, equivalent height of column *h*, and Schofield's pF have been tabulated (Table I).

There are a number of advantages in using suction instead of relative vapor pressure in many situations. The first advantage arises from the nature of the relationship between moisture content and relative vapor pressure. Figure 1 shows that at relative vapor pressures below 0.95 the increase in moisture content is relatively small. This is true for soils as well as many other porous materials. Above a relative vapor pressure of 0.95, however, the increase in moisture content is very large for a small change in vapor pressure. Unfortunately, this is the range that is usually of greatest interest in most soils applications in both agriculture and engineering.

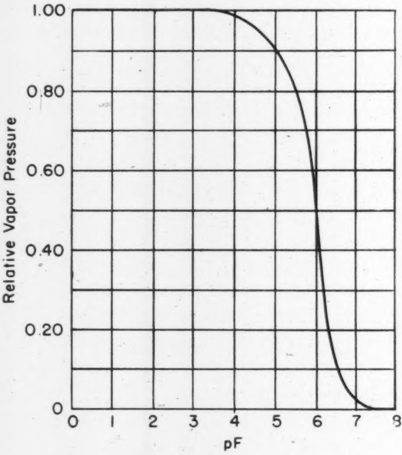

FIG. 2.—Relationship Between pF and Relative Humidity at 20 C.

There are also great difficulties in the accurate measurement and control of relative vapor pressures at the wet end. Since small variations in relative vapor pressure usually correspond to a large change in moisture content, the reliability of vapor pressure measurements above 0.99 is such as to preclude their use. Figure 2, which gives the relationship between relative vapor pressure and suction in terms of pF, clearly shows the expanded nature of the pF scale above 4, the corresponding range of relative vapor pressure being from 0.993 to 1.

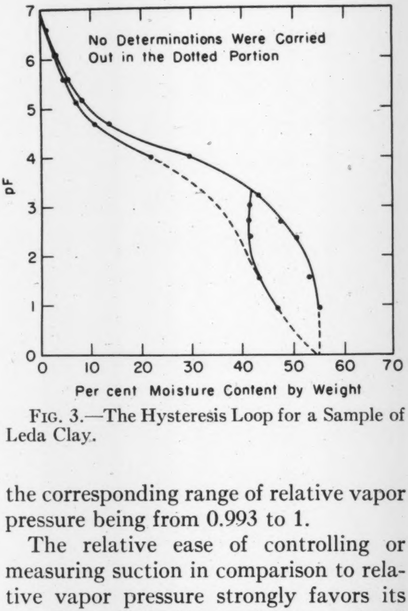

FIG. 3.—The Hysteresis Loop for a Sample of Leda Clay.

The relative ease of controlling or measuring suction in comparison to relative vapor pressure strongly favors its use. This will be discussed in some detail later.

Moisture movement at near-saturation conditions (high relative vapor pressures) is probably largely in the liquid phase. Particularly in soils under natural conditions the movement is attributed to stress gradients in the pore water. The suction h in Eq 3 measured directly describes this stress more conveniently and is not complicated by unrealistic parameters.

A note of caution about the comparison of h measured directly with p/p_0 is in order since the validity of the relationship presupposes a salt-free moisture regime. The quantity h will be affected by salt in held moisture in a different way than p/p_0, but in many cases the differences may be neglected.

HYSTERESIS

The equilibrium moisture content of a porous material obtained at a given relative vapor pressure depends on whether equilibrium is approached by wetting or drying. The effect of this phenomenon is shown in Fig. 3 which gives both the wetting and drying curves. At one time the effect was considered to result from nonequilibrium conditions. It is now known, however, that the sorption process is in a sense irreversible and gives rise to hysteresis effects. The theories advanced to explain hysteresis are discussed in detail by Carman (2). The fact that more than one value can be obtained at the same moisture stress condition must be considered whenever the relationships between suction and moisture content are used.

MEASUREMENT OF SUCTION AT LOW RELATIVE VAPOR PRESSURES

While the concept of suction and its use as a measure of potential can be applied over the entire range of moisture from oven dryness to saturation, different measuring techniques have to be employed for different sections of the range. Relative vapor pressure methods are usually best below pF 4. However, from pF 0 to 4 other methods involving the measurement of suction more directly are usually used.

The suction plate technique for the direct measurement of suction involves placing water in a saturated porous plate in tension, by hanging a column of water from the plate. This may also be accomplished by applying a vacuum to the water in the plate. If this "negative head" is maintained, a sample of soil placed in contact with this "conditioned" plate will establish moisture equilibrium with it. There will be a transfer of water in the liquid phase between the porous plate and the specimen. The time taken to bring the specimen to the same suction condition as the suction plate depends on the rate of water transmission. The range over which the porous plate is operative depends on the pressure difference that can be maintained across the porous plate before it desaturates and passes air and, therefore, is also a function of the maximum size of the pores. When it passes air it no longer operates as a suction plate.

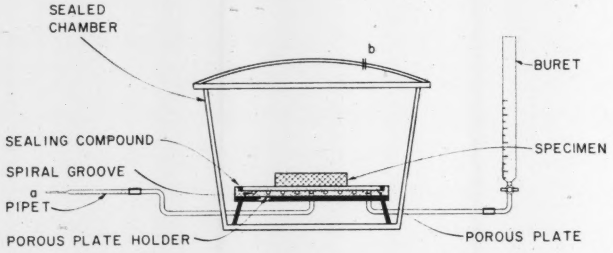

FIG. 4.—Suction Plate Apparatus. Regulated Air Pressure Applied Through Inlet *b*, or Regulated Vacuum at Outlet *a*.

Two methods of maintaining the pressure difference across the porous plate in order to condition it to a given suction have been used and both give essentially the same answer (8). By one method a positive pressure is applied through a pressure regulator to the top of a saturated porous plate (pressure plate) which is held in a metal plate holder. The bottom of the porous plate is in contact with water but open to atmospheric pressure. In the second method the top of the porous plate (suction plate) is kept at atmospheric pressure and the water in contact with the bottom of the plate is placed in tension through a vacuum regulator.

With the types of ceramic porous plates used in this laboratory the suction plate technique is limited to pF's below 3. The apparatus (Fig. 4) is similar to that described by Richards (9) and others with some modifications. In the apparatus shown a rigid suction plate holder made of $\frac{1}{2}$-in. brass is used. A rigid plate holder greatly facilitates making an airtight seal between porous plate and the plate holder and also avoids any volume change between the porous plate and plate holder when the chamber is pressurized. The outside diameter of the porous plate is about $\frac{1}{8}$ in. smaller than the inside diameter of the plate holder. This groove is filled with a suitable water-resistant hard-setting sealer to provide an airtight seal between the holder and the porous plate. A spiral groove on the side of the plate holder facing the porous plate provides a means of water transfer from the sample to the outside of the apparatus. At one end of the spiral, a graduated 1 cu cm pipet is attached and to the other end an ordinary buret is fixed. This provides a means of flushing out any air bubbles when the spiral groove is filled with water. Water is permitted to flow through the system until no air bubbles appear. With a 13-qt capacity pressure cooker the porous plate can be about 10 in. in diameter.

This gives sufficient porous plate area to condition a number of specimens simultaneously. In practice, for soils, small consolidometer rings with a cutting edge have been found useful as sample holders for undisturbed soils. The calibrated pipet attached to the spiral groove is used to determine when equilibrium is reached.

The moisture content of the specimens at various suction levels may be determined gravimetrically by sampling the specimens or the complete specimen may be removed and weighed. In the latter method, the relationship between suction and moisture content is then deduced by finding the weight of oven-dried soil after the final suction determination. This method is subject to a number of errors in practice: the specimen cannot always be removed intact from the porous plate and, once removed, good contact is sometimes not easily achieved between the plate and the specimen.

In a second "suction" apparatus used at this laboratory, which is in many ways similar to apparatus described by Richards (8) and Croney and Coleman (10), the moisture content gains and losses are measured volumetrically. This technique is limited to one specimen at a time but does away with weighing specimens at intermediate suction levels. The apparatus is shown in Fig. 5. The specimen is placed on the porous plate either in the dry or saturated state depending on whether the adsorption or desorption curve is required. Either a pipet or a calibrated capillary tube is used to measure the moisture content change. If large changes in moisture content are envisaged, a long calibrated capillary tube is more convenient. This avoids the necessity of resetting the meniscus at frequent intervals.

The suction or pressure plate technique is used to determine suction - moisture-content relationships in the range from pF 0 to 3. However, as pointed out, suction determinations by the vapor pressure technique are not reliable below pF 4. In the range pF 3 to 4, the "pressure membrane" technique described by Richards (12) and others may be used. The same apparatus shown in Fig. 5 may be used with one important addition. To withstand the large pressure difference required to induce pF's between 3 and 4 without passing air, a membrane with very fine pores must be used, such as Visking sausage skin. In the apparatus shown, the membrane is placed over the porous plate with the sample in contact with the membrane. The walls of the

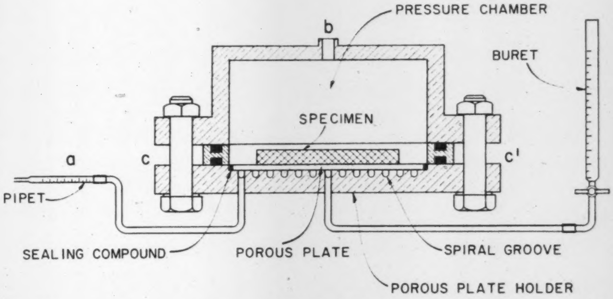

FIG. 5.—Small Suction Plate or Pressure Membrane Apparatus. Regulated Air Pressure Applied Through Inlet *b* or Regulated Vacuum at Outlet *a*.

To Operate as a Pressure Membrane Apparatus Place Membrane from *c* to *c'*.

apparatus are constructed of $\frac{1}{2}$-in. brass sufficiently strong to withstand air pressures equivalent to pF 4. The change in water content of the specimen may again be measured volumetrically or gravimetrically. In practice, the latter has been found more reliable because small air leaks in the membrane frequently invalidate volumetric measurements of moisture content change. The apparatus shown has been found particularly useful since by merely adding the membrane, one apparatus can be used over the entire range from pF 0 to 4.

There are, of course, many other techniques described in the literature for the determination of suction in the low pF range (10). In the author's experience, the techniques described in this paper have proved satisfactory from the point of view of simplicity of apparatus and technique both for undisturbed and prepared specimens. The suction technique may be used with equal facility for rigid and compressible materials. For rigid porous materials such as brick, special care must be taken to provide a good contact between the suction plate and specimen in order to obtain satisfactory rates of moisture transmission.

The apparatus described are for independent methods of measuring relationships between suction and moisture content. The suction technique is also used frequently as a means of controlling the moisture status while other properties are being measured. To do this, the suction technique is incorporated into the design of larger apparatus where the parameters to be studied are a function of the moisture status. This can be an invaluable asset in many types of experiments with porous materials. Two general approaches are possible: one may either measure the variation in suction as the experiment progresses or impose a definite suction throughout the experiment. The exact methods involved are a matter of adapting the suction technique to the apparatus being used.

EFFECT OF TEMPERATURE ON SUCTION MEASUREMENTS

In the low suction range, the effect of temperature change on suction is due mainly to surface tension variation with temperature. It is assumed that the establishment of the equilibrium is through the liquid phase. Under these conditions, variation is approximately 0.1 pF units for a 10 C change in temperature, according to Croney and Coleman (11), provided the proper precautions are taken. A further difficulty, owing to temperature fluctuation, arises from condensation on the inside surfaces of the apparatus. At this laboratory, suction plate, pressure membrane, and unsaturated permeability determinations are carried out in a small temperature-controlled room where temperature variations do not exceed ±0.1 C of control point.

SOME APPLICATIONS OF THE SUCTION CONCEPT IN SOILS

Shear Strength of Unsaturated Soils.—The well-established concept of effective stress for saturated soil

$$\sigma' = \sigma - u \text{.............} (5)$$

where:

σ' = effective normal stress,
σ = applied normal stress, and
u = pressure in the pore fluid,

has recently been extended to unsaturated soil by Australian workers (13–15). This equation has been rewritten in the form

$$\sigma' = \sigma + p'' \text{............} (6)$$

where the quantity p'' is the suction (that is, negative pressure) of the soil moisture and is substituted as a positive quantity. According to Terzaghi, the effective stress is that part of the total stress which produces measurable effects such as consolidation or an increase in shearing resistance. This means that these "effects" can be attributed in part to suction; this has already been demonstrated with reasonable certainty by the Australian workers.

Croney and Coleman (**10**) suggest that for saturated compressible clays, the pressure void ratio curve determined by the consolidation technique is in fact a suction moisture content drying curve. At any particular applied pressure in the consolidometer the pressure, p, is equal to the effective stress, σ', at equilibrium since the pore pressure, u, is 0. A similarity between consolidation curves and suction - moisture-content curves determined by the suction plate and pressure membrane techniques was the basis for the Australian workers concluding that, for compressible clays, the concept of effective stresses extended to pore pressures which are truly negative with respect to free water at atmospheric pressure. Noncohesive soils did not respond in a similar way beyond the range where the soil pores emptied under tension forces.

The shear strengths of unsaturated soils is obviously of great importance for Australian conditions. In a recent paper, Aitchison (**15**) concludes that the variation of suction in the pore water for the Australian environment appears to be a major determinant of soil strength in cohesive soils.

Equilibrium Moisture Distribution in Soil Above a Shallow Water Table:

The moisture changes that occur in the soil underneath airfield pavements, etc., subsequent to construction has been extensively studied by the British Road Research Laboratory (**16–19**). The premise is that subsequent moisture changes may invalidate the design based on soil strengths determined before construction. It appears that the main determinants of the equilibrium moisture conditions above a constant water table are embodied in the following relationship:

$$\alpha P = s + u \qquad \text{(7)}$$

where:

P = normal pressure,

s = the moisture suction of the soil,

u = the pore water pressure, and

α = the fraction of the normal pressure that is effective in changing the suction.

The relationship apparently is most easily determined for the case of an impermeable surface cover. To determine the equilibrium moisture-content distribution, u is either measured with tensiometers or calculated from the location of the ground water table. P is calculated from the total weight of the wet soil above the point in question, and α can be evaluated from loading and shrinkage tests. Thus s can be calculated and, from suction - moisture-content curves, the estimated moisture content is obtained. Research workers who developed this method report good success with it; however, to what extent the method has been generally accepted by others is not known. There is no question of the usefulness of predicting, in advance of construction, the moisture content changes that will occur under structures.

Unsaturated Moisture Flow:

Moisture flow in the liquid phase for unsaturated soils follows Darcy's law, $v = ki$, under isothermal conditions. In contrast to saturated flow, k is not a constant but is a function of the average moisture suction. For example, if the levels of suction are 10 and 20 cm separated by a distance l, the velocity of flow will be different than when the levels of suction are 190 and 200 cm, again separated by distance l, although the gradients are the same. To a large extent the variation of k depends on the state of saturation. The greatest variation occurs in uniform sands or silts which have a critical suction where almost complete desaturation occurs. At suctions greater than this, the liquid phase through the pore system is discontinuous, and the greatly reduced flow is through the adsorbed phase.

determined in the laboratory (21,22). Nevertheless, the concept of suction potential as a driving force has contributed to the understanding of unsaturated flow in nature. This type of flow is thought to account for the distribution of water at equilibrium above the shallow water table as discussed in the previous section.

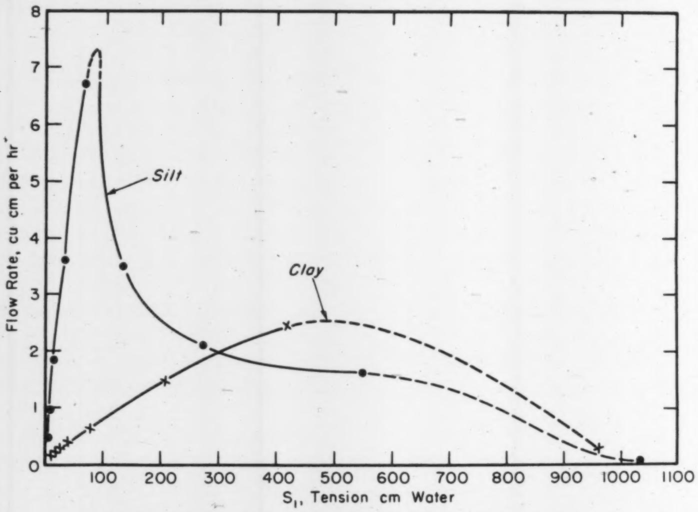

FIG. 6.—Flow Rate as a Function of S_1 with S_2 Held at Zero.

Preliminary experiments with an unsaturated permeability apparatus reported previously by the author (20) compare flow rates below pF 3. The flow rates shown in Fig. 6 were obtained by holding the suction S_2 at one end of the specimen at zero and increasing the suction at the other end S_1 in small increments. These results show familiar characteristics of unsaturated flow. At low tensions the flow rates are higher in silt than in clay, but this reverses at higher tensions. At present no satisfactory method for predicting unsaturated flow with any degree of certainty is available. As a consequence, it is usually

Suction in Relation to Frost Heaving:

The phenomenon of frost heaving as related to the performance of shallow foundations or structures, roads, and airports is still not completely understood. Much of the present knowledge stems from the early work of Taber (23) and Beskow (24). More recently Haley et al (25), Ruckli (26), and Jumikis (27) have made notable contributions. The author (20, 28–29) has recently studied the mechanism of ice lensing in the laboratory using artificial and natural soils. The concept of suction has been useful in studying the equilibrium pressure relationships in the ice-water-soil system and has led to the development of a tentative theory of ice lensing in soils.

The driving force for moisture movement is the suction developed at the ice-water interface. This force is the most important feature of frost heaving since it must exist for ice lenses to form. Experiments show that the suction has increased to a maximum in a closed system when the ice lens growth stops, that is, at equilibrium, but it is greatly influenced by the dimensions of the pores and the overburden pressure. The thermodynamic equilibrium conditions have been described based on a simple model of the ice-water interface in a small element of the granular system.

Electrical Resistance Block Method of Measuring Moisture in Soil:

A reliable nondestructive method of measuring soil moisture content in the field has still not been perfected, but recent developments with the neutron meter show promise. The electrical resistance block technique of measuring moisture is discussed here partly because it is still used in practice and also because this technique demonstrates rather an important principle as follows. The equilibrium moisture condition between dissimilar porous materials exists when the suctions are similar although the moisture contents may vary widely. The electrical resistance meters made of porous materials are therefore more appropriately referred to as suction meters.

Earlier workers noted that the electrical resistance of soil was a function of its moisture content. Investigations revealed that soil density, particle-size distribution, mineral type, and salinity of the soil solution influence resistance sufficiently to limit its usefulness as a measure of moisture content. To overcome these difficulties the electrodes were buried in a porous block which was then placed in contact with the soil. In this way it was hoped to limit the variation of resistance to moisture content only.

In one method of calibration the resistance meter is buried in a block of saturated soil selected from the site where the meter is to be installed. The soil is then dried in stages and allowed to come to equilibrium. The electrical resistance is measured at each equilibrium state, and the soil is sampled for moisture content. In this way a complete calibration for the drying cycle can be obtained. This calibration, however, applies to only the one soil used in the calibration.

In this laboratory a calibration was carried out to establish the direct relationship between suction and electrical resistance of the moisture blocks. The suction of the meters was controlled using suction plate and pressure membrane techniques, and electrical resistance readings were taken when suction equilibrium was established. If the further relationship between suction and moisture content for the soil is determined, all the information is available to convert electrical resistance readings of the meters to moisture content of the soil with which it is in suction equilibrium. The commercial moisture meters that did not have a flat contact surface were embedded in plaster of Paris. In this way, the same method of calibration could be used for all the meters irrespective of type.

In practice, the electrical resistance method of measuring the moisture content of soils is beset with many difficulties. These are outlined in some detail by Penner et al (**30**).

CONCLUSIONS

The concept of suction has been usefully employed both in practice and as a research tool. The suction characteristics of a porous material are in essence sorption isotherms, but the interpretation of the phenomena has led to new methods of determining and expressing the affinity of a porous material for water and is particularly useful in the high relative vapor pressure range. In this region the water is held largely in the macropore system. The concept of suction and its method of determination provides a realistic approach to many problems in soil engineering.

Acknowledgments:

The author wishes to express his appreciation to Mr. N. B. Hutcheon for his guidance in preparing this paper and to Mr. R. F. Legget for his continuing interest in moisture problems, and to Mr. J. M. Kuzmak, who designed the apparatus shown in Fig. 5 of this paper.

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THE MECHANICAL BEHAVIOR OF CHEMICALLY TREATED GRANULAR SOILS*

By Robert L. Schiffman[1] and Charles R. Wilson[2]

Synopsis

This paper is the result of a laboratory experimental research program on the mechanical behavior of chemically treated granular soils. The chemical used is commercially known as Stabilizer AM-9 and is manufactured by the American Cyanamid Co. The granular soils were selected with respect to mean grain size and range of grain sizes.

The research was concentrated on three phases of behavior:

1. The mechanical strength through unconfined compression and triaxial compression tests of the saturated soil when freshly treated with a constant amount of chemical. The variables studied were mean grain size, range of grain sizes, and the relative density of the soil. Results are presented in terms of significant soil variables and the shear strength relations of Coulomb and Mohr.
2. The unconfined compression strength of a series of soils in relation to results of the effect of monomer concentration.
3. The unconfined compression strength of a single soil in the dense state in which the effects of wetting and drying were investigated. This phase included an investigation of the strength increase of this soil with aging.

The research program was conducted in several steps: (*a*) the establishment of a theoretical hypothesis of granular soil behavior when treated with an addition polymer in relation to the mechanisms of soil-chemical action and the selection of soil properties influencing soil strength; (*b*) the establishment of a statistical hypothesis and the detailed planning of laboratory control experiments, in view of the number of experiments to be performed, level of experimentation, and repeatability; (*c*) a description of the laboratory experimental procedure, and the results of the experimental program based on observations of relations between soil and chemical variables; and (*d*) a report of the authors' interpretations of the mechanical action of chemically treated granular soils based on data for Stabilizer AM-9 and further extended to any addition polymers.

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GENERAL

The use of chemicals for soil stabilization offers one of the potentially most fruitful means of soil modification. Other methods of stabilization in extensive successful use at present may be limited in effectiveness, since basically they rely on the improvement of the existing soil structure and do not alter the inherent nature of the soil. Chemical stabilization, however, may alter the basic existing soil properties as well as improve them (**1**).[3] Chemical soil stabilization thus serves a dual purpose: (*a*) improvement of existing soil properties, and (*b*) fundamental alteration of the existing soil chemistry to produce a new soil.

The action of chemicals on soil can be separated into three types:

1. The use of so-called trace chemicals. This type of chemical reacts with the soil elements to form a new soil derived from a more desirable mineral. An example is that of ion- or base-exchange chemicals. Small amounts of lime when added to a sodium soil will replace the sodium ions and produce a more stable soil. Similar modifications have been produced by the use of trace quantities of dispersing agents such as sodium polyphosphate and calcium lignosulfonate.

2. The action that chemicals can produce in a soil as an inert void filler. An inert chemical that does nothing more than fill the voids of a soil mass will place mechanical constraints on the deformation of individual particles. These constraints, in turn, will alter the phenomenological response of the soil mass to mechanical forces. Silicates and chrome-lignin partially produce this type of action.

3. The chemical action of adhesion or cementation. The chemical forms a bonding link, thus creating forces connecting soil particles. In terms of individual grains within a soil mass, these forces are external to the grain and act to hold grains together. In terms of the entire soil mass, these forces are internal restraints strengthening the entire system. Chemicals whose primary action is of this type include calcium acrylate and American Cyanamid Stabilizer AM-9.

Most chemicals of the two latter types act in some degree both as void fillers and as bonding agents.

The response of a chemically stabilized soil mass to imposed external mechanical forces depends on the type of chemical action and on the geometrical configuration of the soil-chemical matrix. Where the chemicals are used in trace amounts to alter the physicochemical nature of the soil, the responses to mechanical action are those of the altered soil and are not directly related to the response of the chemical. When the chemical forms an inert void filler, the response to imposed external forces is basically that of the soil but modified in accordance with the geometric restraints imposed by the void filler. Types of chemical stabilization which impose physical force restraints on the individual grains exhibit a mechanical response that is basically that of the soil, but in this case is modified by the force restraints of the chemical. The configuration of the soil-chemical matrix determines the mode of mechanical action for the two latter types of chemical stabilization. In an open-structured system where the grains are basically unconnected, the mechanical action is that of the chemical, with the soil grains forming a mass-point system in a chemical matrix (**2**).

Close-structured systems, in which the soil particles are in intimate contact, behave basically as the particles behave, modified only by the restraints established by the stabilizing agents.

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[3] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 1244.

Stabilizer AM-9 is an addition polymerizing chemical which when treated with a redox catalyst will form a gel. The action of this gel is twofold: (1) It reacts with the surface of granular soil grains, adhering to the grains and binding separate grains, thus establishing internal force restraints in the system and strengthening the entire soil-chemical complex; and (2) it fills the voids of the granular soil mass, imposing geometric restraints on the entire system.

This paper is concerned with the mechanical behavior of granular soils when treated with an addition polymerizing chemical. The purpose of the investigation was to determine experimentally the effects of this type of chemical on the strength and stress-strain-time behavior of granular soils. In particular, quantitative data were obtained on the properties of granular soils treated with Stabilizer AM-9.

Certain particular influences of stabilization were studied: (*a*) effect of relative density on strength; (*b*) effect of mean grain size and grain size distribution on strength; (*c*) effect of chemical concentration on strength in newly stabilized conditions; and (*d*) effect of aging, including wetting-and-drying cycles, on strength.

Well-documented investigations into the behavior of granular soils has proven that the mechanical properties of these materials are governed by the following (3):

1. *Mean Grain Size of the Granular Mass.*—The grain size measure of the mass for this study is that size for which 50 per cent by weight of all sizes is finer. It will be referred to as D_{50} and is expressed in millimeters.

2. *Average Grain Shape of Granular Soil Mass.*—The average grain shape is divided into four classifications which establish broad limits of angularity. These are: angular, subangular, subround, and round. The shape of the grain determines the ability of the soil to pack and the amount of surface contact available between grains. The coupling of these two measures results in the conclusion that the higher the degree of angularity the lower the achievable unit weight and the lower the maximum strength (4).

3. *Variation of Grain Size of a Granular Mass.*—Soils have finite size variations, and thus the deviation about a mean size can be expressed in terms of the range of sizes present in a given distribution. The ability to pack is a function of the variation of grain size. A uniformly sorted soil will have the least potential maximum unit weight and strength, while a soil with a continuous spectrum of all sizes will potentially have the highest possible unit weight and strength. All granular soils will fall somewhere between these two extremes.

4. *Shape of Grain Size Distribution Curve.*—The shape of the size distribution function is an additional measure of the central tendency of the grain size distribution and is measured by the skewness of the mode size with respect to the mean size. The maximum attainable unit weight and the maximum obtainable strength are directly related to the shape of the grain size distribution curve.

While the above four variables will in general control all granular soil behavior, the range of behavior within a given distribution and particle shape is defined by the relative density of the given granular soil (5).

The deformation response of a granular soil to external boundary tractions or displacements can be broken down into two basic modes of action. The first and most important is a macroscopic grain slip without any deformation of the grain. This slip is a nonrecoverable rigid body movement caused by an unbalance of the external forces on given grains. The second mode of action occurs subsequent to the rigid body motion of the grains and constitutes a deformation of the given grains. This deformation has an elastic recoverable portion and an inelastic nonrecoverable one. The slip phenomenon is frictional and is controlled by the configuration of the grain contacts as reflected by the relative density and the amount of contact, determined by the grain size, grain shape, and the distribution of grain sizes. The general character of the deformation mechanism is indicated by the volume change that takes place during compression, as well as the stress-strain relations (**6**).

The strength of a granular soil mass is quite adequately described by a linear failure envelope through the origin, as defined by Mohr and Coulomb (**7**).

MECHANICS OF STABILIZATION

The deformation and subsequent failure of a granular soil mass depends upon the nature of the net Coulomb friction between grains and the magnitude of the compliance forces between grains. An inert gel filler will impose restraints to movement which will increase the tangential force required to accomplish grain slip.

The angle of internal friction between soil grains is dependent upon the compliance forces between grains and the character of the contact surface. If a filler material could be forced into the void volume without coating the grain contacts, the net effect would be to increase the stresses required to impel movement and there would be an increase in the measured angle of internal friction. The introduction of stabilizer into the soil coats the grains and modifies the nature of the friction resistance between grains. The net effect will be to increase the compliance forces but decrease the molecular friction forces resisting slip. Loose granular soils when stabilized with relatively weak polymers should exhibit a marked decrease in the angle of internal friction due to the lubrication effect of the polymer coating on the grains.

The shearing of dry dense granular soils is always accompanied by an initial volume increase prior to failure. In order to achieve slip the entire mass must expand and place the particles in a condition amenable to friction slip. The measured shearing strength of the soil is largely controlled by the forces inducing a volume change to a state compatible with slip failure. Thus the angle of internal friction of a dense granular soil is not solely the measure of the forces required to move the granular surfaces across each other but is also the measure of the forces required to expand the mass until the particles can slip across each other. The influence of a weak polymer on the dense soil should not materially change the measured angle of internal friction.

A granular soil without stabilizer is cohesionless, and in the completely dry state will not exhibit any internal forces. A chemically stabilized soil will exhibit marked internal forces, immediately indicated by its stability to molding. These forces are the internal tensions established by the gel adhering to the soil particles.

When a soil treated with a weak gel is permitted to age, the gel will shrink. The internal mass forces of the soil are greater than the tensile strength of the polymer, and in the resulting action of drying the gel itself will crack or fail in tension. The new surfaces formed by the crack will move toward the grain contacts, forming capillary menisci. The presence of these menisci will produce additional internal tensile stresses between grains, thus greatly enhancing the compressive strength of the soil mass.

If the soil was saturated with water after drying, the capillary forces would be relieved, producing a stabilized mass with a reduced strength due to the loss of some of the internal restraints.

EXPERIMENTAL PROGRAM

Design of the Experiment:

The experimental program was designed to evaluate the following influences:

(*a*) The dependency between the relative density and the shearing strength of a freshly stabilized granular soil;

(*b*) The dependency between the mean grain size and the shearing strength of a freshly stabilized granular soil;

(*c*) The dependency between the range of grain sizes and the shearing strength for freshly stabilized granular soils;

(*d*) The failure law governing freshly stabilized granular soil;

(*e*) The influence of amount of stabilizer on the unconfined compression strength of a freshly stabilized granular soil;

(*f*) The influence of aging on the unconfined compression strength of a given stabilized granular soil; and

(*g*) The influence of cycles of alternate wetting and drying on the unconfined compression strength of a given stabilized granular soil.

In order to develop the maximum efficiency from the test data, the experiment was designed to be run on a statistical basis (**8**).

There are two bases for the statistical design of experiments. The first basis is the statistically undefined experiment in which the laboratory program aims at defining the mechanical behavior over an extended area. In this type of test, some of the more elegant statistical methods, such as sequential analysis and factorial design are of little value. The most efficient statistical design was a regression analysis to define the mean behavior as a functional relationship between the variables concerned, taken two at a time. The total behavior, as distinguished from the mean response, was ascertained from statistical testing of small sample theory.

It was implied in the experimental design that each experiment was an independent event taken from a normal population. The regression analysis was based on a presumption of linearity between tested response, and in each case the correctness of the linearity assumption was tested. In extending the results for a finite sample size to that of the population, the chi-square test was used for the variance and Students' *t*-distribution was used for the mean values.

For the wetting-and-drying experiment a different statistical design was used. For this part of the program it was necessary to determine the interac-

TABLE I.—FACTORIAL WETTING AND DRYING EXPERIMENT.

NOTATION FOR D_j[a]

Index, *j*	Drying Level
7	30 days dry—15 days soaked
6	15 days dry—15 days soaked
5	10 days dry—10 days soaked
4	5 days dry— 5 days soaked
3	3 days dry— 3 days soaked
2	1 day dry— 1 day soaked
1	0 days dry— 0 days soaked

NOTATION FOR C_p[b]

Index, *p*	Number of Cycles
1	1 cycle of drying and wetting
2	2 cycles of drying and wetting
3	3 cycles of drying and wetting

[a] D_j = Drying level.
[b] C_p = Cycling level.

tions between length of time of drying and the number of cycles of wetting and drying in terms of their influence on the unconfined compression strength. The measurements were based on the ratio of the strength of the stabilized soil after a given treatment, σ_t , to the strength in the freshly gelled state, σ_0 . Thus the response variable was

$$Y = \sigma_t/\sigma_0 \ldots\ldots\ldots\ldots\ldots (1)$$

This experiment was designed as a factorial experiment and analyzed by techniques of variance analysis and F-distribution testing to determine if interactions between variables were significant.

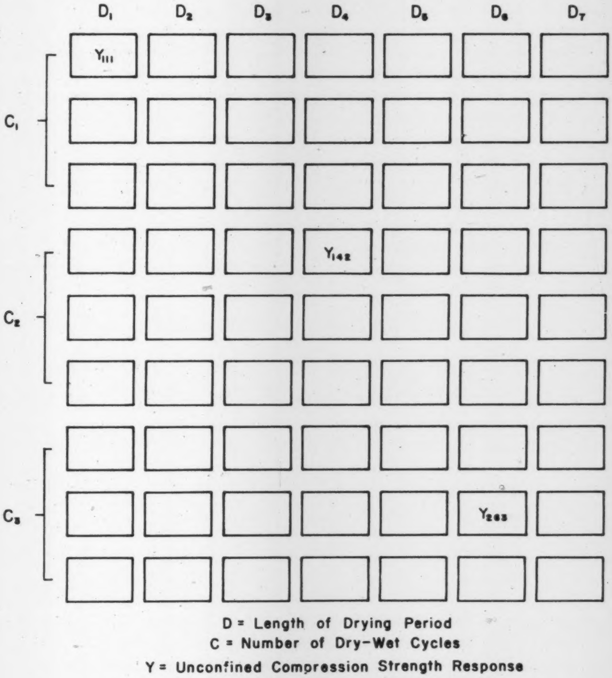

D_1
D_2
D_3
D_4
D_5
D_6
D_7
C_1
C_2
C_3
Y_{111}
Y_{142}
Y_{263}
D = Length of Drying Period
C = Number of Dry-Wet Cycles
Y = Unconfined Compression Strength Response

Fig. 1.—Dry-Wet Factorial Design.

The wetting-and-drying experiment was performed on a single soil. In order to account for randomness, an order of replication of three was arbitrarily established.

As shown in Table I, there were seven levels of wet-dry time and three levels of cycles of wetting and drying. The complete layout for the experiment is shown in Fig. 1. In order to test the interactions between the length of drying and wetting and the number of cycles of wetting and drying, with a replication of three, 63 individual experiments were required. This number was over and above the number of control experiments required to determine the freshly gelled response.

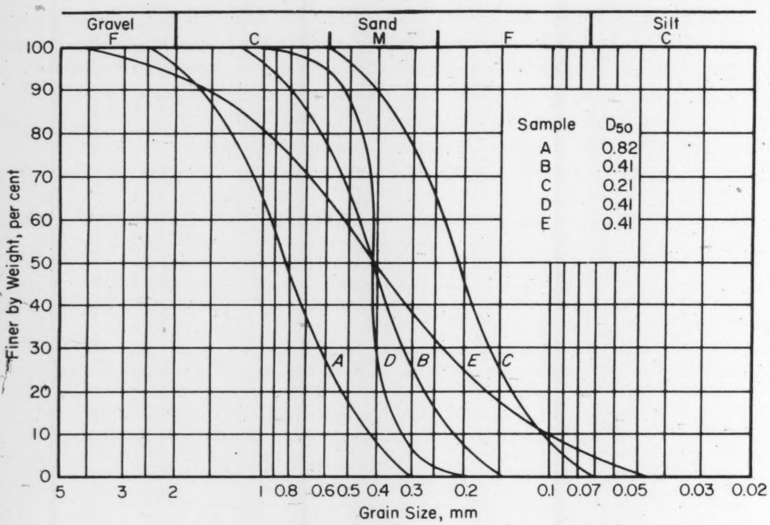


FIG. 2.—Grain Size Distribution of Experimental Soils.

TABLE II.—GRAIN-SIZE DISTRIBUTION OF SELECTED SOIL SAMPLES, SIEVE PROPORTION, PER CENT PASSING.

	Sieve No. 4	Sieve No. 8	Sieve No. 16	Sieve No. 30	Sieve No. 50	Sieve No. 100	Sieve No. 200
Grain Size, mm	4.76	2.38	1.19	0.59	0.30	0.15	0.074
Sample A	...	25	50	25	...	...	...
Sample B	...	...	25	50	25	...	...
Sample C	...	...	...	25	50	25	...
Sample D	...	...	5	90	5	...	...
Sample E	5	10	23	24	23	10	5

Material Properties:

The soils obtained from local suppliers for testing purposes were a mixture of two river deposit materials, composed mostly of silicates. The soil variables were selected as being representative of a wide range of granular soils found in nature. The natural soils obtained were hand-sieved and combined in the manner shown in Fig. 2 and Table II. The soil descriptions are in accordance with the Burmister Identification System (3). The soil particles were classified as subround. The soils used were relatively clean and sterile. A mineralogical analysis was performed by members of the Geology Department of Lehigh University. The results of this analysis are presented in Table III. The basic unit weight and grading properties of the test soils are presented in Table IV.

The range of behavior of the apparent angle of internal friction – relative density relations was established on the basis of data from other investigators (**4,9,10**). In the loose state the total range of soils had angles of internal friction which varied between 28 and 34 deg and 43 to 48 deg in the dense state.

In all cases, the chemical used was Stabilizer AM-9 in varying percentages by weight, along with the catalyst system of ammonium persulfate and sodium thiosulfate. Table V shows the quantities of chemical used to make up solutions of the stabilizer for the varying concentrations used.

Although the weight of Stabilizer AM-9 for a given concentration was fixed with respect to the total solution, the stabilizer-water ratio varied with differences in the amount of catalyst. The percentage change was of such a minor nature that no significant differences due to change in the amount of catalyst were detected in subsequent analysis.

TABLE III.—MINERAL ANALYSIS OF SOIL.

MINERAL	BY VOLUME, PER CENT
Quartz	54
Plagioclase	33
Microcline	3
Chlorite	4
Augite	2
Heavy minerals[a]	4
	100

[a] Hornblende, magnetite, ilmenite, garnet, biotite, dolomite, etc.

TABLE IV.—GRADING AND DENSITY PROPERTIES.

Soil Sample	Maximum Dry Unit Weight, lb per cu ft	Minimum Dry Unit Weight, lb per cu ft	Range of Grain Sizes, C_R	D_{60}, mm	Specific Gravity
A	109.0	91.0	1.53	0.82	2.68
B	110.5	91.0	1.53	0.41	2.68
C	112.0	91.0	1.53	0.21	2.68
D	103.0	86.0	0.59	0.41	2.68
E	120.0	100.0	2.85	0.41	2.68

The AM-9 monomer, being chemically stable over a period of several weeks, was prepared in relatively large quantities. During preparation, it was observed that a quantity of undissolved residue remained in suspension. The monomer used in sample preparation was filtered through cloth in order to remove the residue.

Experimental Procedure:

The experimental procedure was designed to test the several variables in such a manner that good repeatability of experimental results could be obtained.

Since relative density was one of the prime factors investigated, it was necessary to mold the specimens of soil at various degrees of compactness. In order to eliminate local failures due to pockets of material of a density condition differing from the over-all sample, it was necessary that each individual sample have the same degree of compactness throughout its volume.

TABLE V.—MIXING PROPORTIONS.

Stabilizer Concentration, per cent	Stabilizer AM-9, g	Catalyst, g	Water, g	Total, g
LOOSE STATE				
5	5.0	1.2	93.8	100
7	7.0	1.2 (0.7)[a]	91.8 (92.3)[a]	100
10	10.0	1.2	88.8	100
15	15.0	1.2	83.8	100
25	25.0	1.2	73.8	100
DENSE STATE				
5	5.0	1.0	94.0	100
7	7.0	1.0 (0.7)[a]	92.0 (92.3)[a]	100
10	10.0	1.0	89.0	100
15	15.0	1.0	84.0	100
25	25.0	1.0	74.0	100

[a] Differences in catalyst percentages used in different test series.

In order to assure replication, special molds were constructed. Each mold consisted of a 3-in. diameter split Lucite tube held together by brass stud bolts clamping top and bottom Lucite plates. A detachable collar was provided in order that the prepared specimen could be 2 in. longer than desired. This device eliminated variations in relative density at the top of the specimen.

The test specimens were 3 in. in diameter and 8 in. long.

The first step in the preparation of specimens for the dense state was to pour approximately 2 in. of solution into the assembled mold. Next, the soil to be compacted was placed in the mold to a depth of about ½ in. with care, so that there were minimal air voids in the sand-solution mixture. The bottom layer of soil was then compacted by vibration using a Burgess Vibro-Tool fitted with a 2-in. diameter foot. The Vibro-Tool was applied by the operator based on results of a large series of preliminary compaction tests. This process was continued by the additions of solution and ½-in. layers of soil until the level of the soil in the assembled mold was at least 1 in. above the predetermined level of the trimmed specimen.

Formation of the loose state was started by pouring approximately 2 in. of solution in the assembled mold. Soil was then gently and slowly dropped into the mold from the top. This was done, either by means of a funnel, held just above the level of the solution, or by slowly shaking soil from a spoon held over the top of the mold. With either method, the level of the solution was kept continuously about 2 in. above the level of the soil. The addition of fluid and soil was continued until the level of the specimen was at least 1 in. above the final height of the specimen.

Specimens to be compacted in intermediate relative density states were first formed in the loose condition. The desired relative density was then obtained by imparting impact energy to the assembled mold and its contents. This energy was induced by blows from a wooden mallet applied evenly in number and intensity to both the base and the collar plates of the assembled mold. The degree of relative density was controlled by the number and intensity of the applied blows. While it was realized that the above procedure would not guarantee a specimen at any particular relative density state, or even in a state of uniform unit weight, enough tests were performed to enable the recognition and subsequent discarding of specimens that failed in localized regions of the specimens.

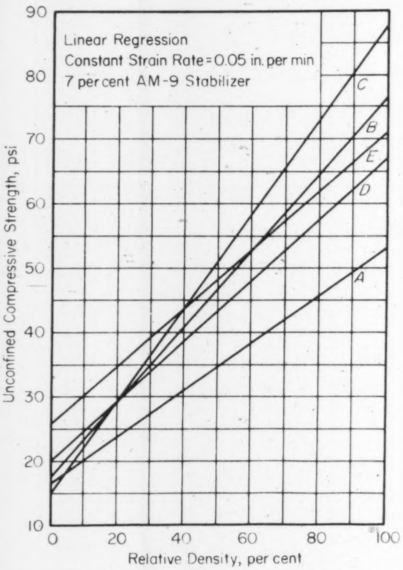


FIG. 3.—Unconfined Compressive Strength-Relative Density Relations.

The method employed to achieve the intermediate relative density states was less than satisfactory, as indicated by the relatively large number of discarded specimens. When the effect of relative density was proved to be linear, all subsequent tests were run at the relative density extremes.

A large number of preliminary tests was run to develop techniques that would achieve a condition of complete saturation. By utilizing these techniques, the saturation obtained for all test specimens was between 97 and 100 per cent.

All compression tests were performed on a Tinius Olsen Electomatic Universal testing machine. Prior to testing, the machine was calibrated for accuracy of the weighing system and precision of strain rates. In the region of the applied loads, the machine was found to be accurate to $\frac{1}{4}$ lb or within 0.05 per cent of the lowest ultimate load. The precision of the strain rates used was within 1 per cent of the rated velocity. The compression tests were all strain-controlled at a deformation rate of 0.05 in. per min. A pilot series of unconfined compression tests indicated that between 0.025 and 0.10 in. per min there were no significant differences in the strength of the soil with respect to strain rate.

TABLE VI.—STATISTICAL LINEAR REGRESSION ANALYSIS OF UNCONFINED COMPRESSION STRENGTH *versus* RELATIVE DENSITY.

Soil Sample	S_E, psi	m, psi	Confidence Limits on m	σ_E, psi	Confidence Limits on σ_E, psi	Correlation Coefficient. r
A.......	1.95	1.29	0.95	3.56	0.95	0.985
B.......	3.04	2.14	0.95	5.75	0.95	0.965
C.......	3.48	2.62	0.95	6.94	0.95	0.992
D.......	3.08	1.94	0.95	5.43	0.95	0.994
E.......	2.71	2.04	0.95	5.39	0.95	0.987

S_E = Standard deviation of the strength based on the test regression line.

m = Variation of the population regression line from the test regression line within the confidence limits set.

σ_E = Standard deviation of the population based on the population regression within the confidence limits specified.

The strength testing for the freshly gelled test series was completed within 2 hr after specimen preparation.

In aging and drying, the specimens were mounted on racks in the laboratory. The laboratory was air conditioned with commercial air conditioning units which maintained the temperature at a mean of 75 F and the relative humidity at a mean of 52 per cent.

MECHANICAL BEHAVIOR

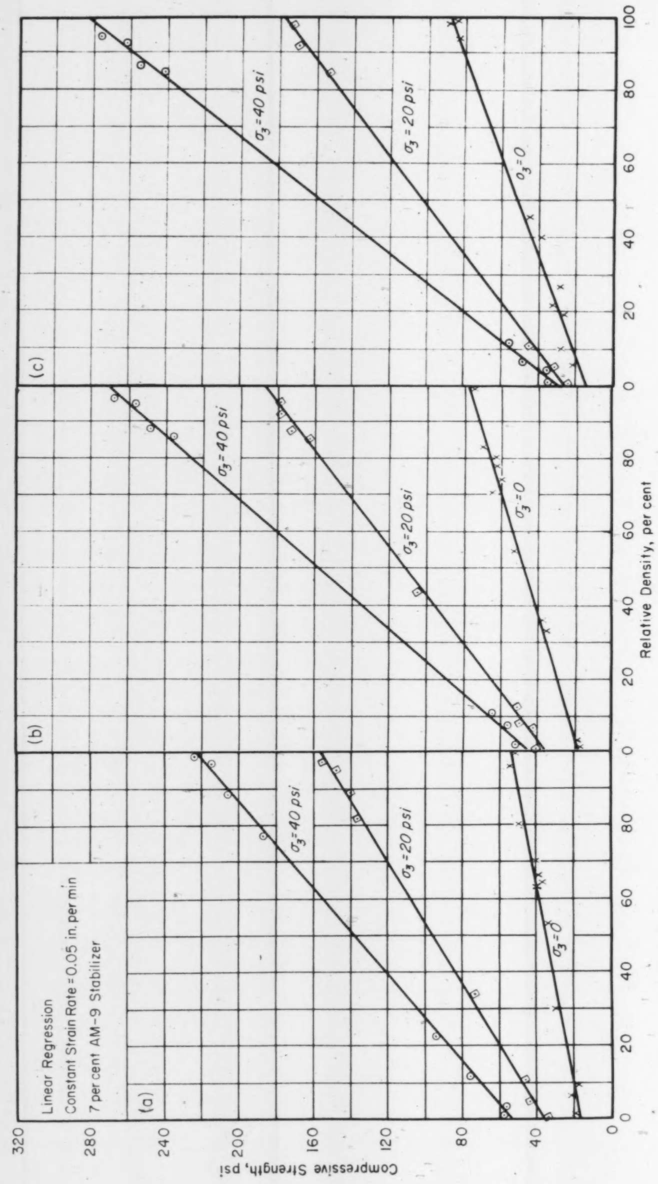
Influence of Soil Properties:

All five soils were studied to investigate the influence of grain size, range of grain sizes, and relative density. The monomer concentration was maintained at 7 per cent. While strength control was mostly based on the unconfined compression tests, a series of triaxial compression tests was run for the exclusive purpose of developing a failure criterion.

The influence of the soil properties on the unconfined compression strength was based on a total of 56 usable tests taken on each of the five soil samples.

The theoretical hypothesis regarding the relation of shearing strength and relative density stated that a significant functional dependency existed. This hypothesis was verified by the analysis of the test results. In the first instance the linearity was in question. A third degree polynomial was fitted to the unconfined compression test – relative density data. The only significant terms in the resulting expression were the linear terms. Tentatively it was decided to fit a linear regression line to the data and to determine the statistical confidence limits for this fit.

Figure 3 presents the least square regression line for the unconfined strength – relative density relation for each soil. The values in Table VI are the statistical entities for this regression analysis. The fact that the correlation coefficient is close to unity is further indication of the linearity of the relative density relationship. The regression lines shown in Fig. 3 are actual computations of a mean value. The deviation about this mean for any relative density considering the population of tests would be a band of 2M psi about the regression line. This

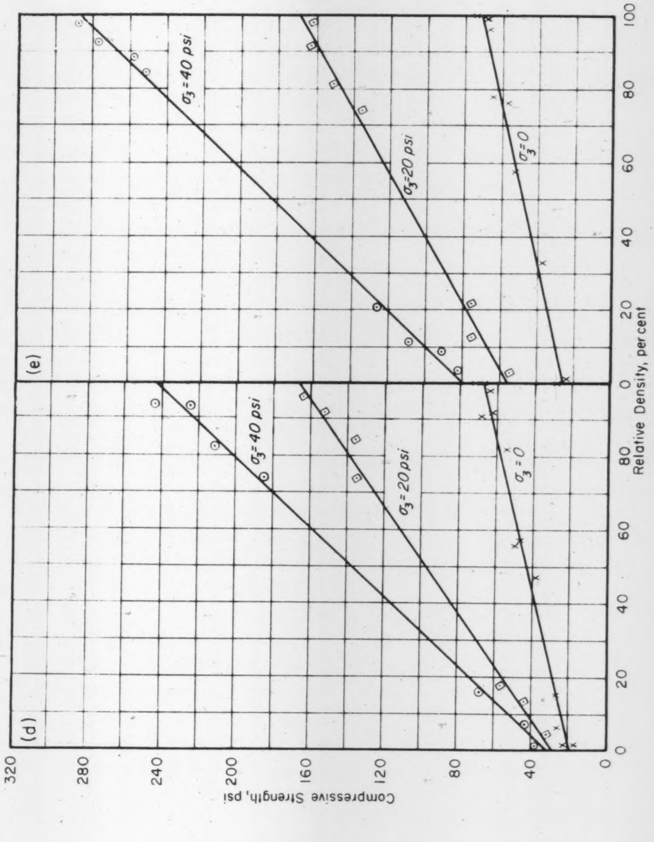


(a) Soil A

(b) Soil B

(c) Soil C

FIG. 4.—Strength-Relative Density Relations.



(*d*) Soil D

(*e*) Soil E

FIG. 4.—*Continued.*

band will occur for 95 per cent of all possible observations. Any individual test out of the entire population of tests will fall within a range of 2 σ_B on either side of the mean line, 2M. This will occur 91 per cent of the time. As an example, considering soil A at 50 per cent relative density, the mean tested strength is 39 psi. Extending this to 95 per cent of any future tests, the mean range will be from 37.71 to 40.29 psi. Ninety-one per cent of all tests will fall within a range between 30.59 and 47.41 psi.

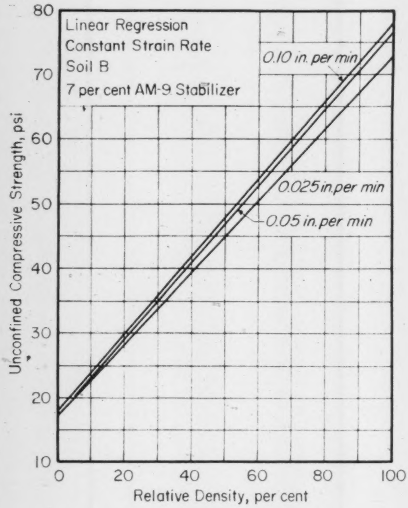


FIG. 5.—Strain-Rate Influence.

Although a regression analysis was performed on the triaxial tests results, the number of tests was not sufficiently large to complete a usable statistical analysis. The regression was based on an assumption of linearity. Since the phenomenon being studied remained constant and since the relationship at one extreme of lateral pressure was proved to be linear, it is logical to presume linearity throughout the entire range.

Figures 4(*a*) to (*e*) present the data and the regression lines for each test series. These curves indicate that the slope of the strength – relative density relation varies with changes in the lateral pressure. The magnitude of this variation is so great as to be outside the statistical limits of behavior. An additional significant fact is that the slope increases with increasing lateral pressure. A third factor is the general closeness of results in the zero per cent relative density state for soils A, B, C, and D. This is indicative of the fact that the change in behavior of loose stabilized soil is less dependent on soil type and lateral pressure than that of unstabilized soil. Thus apparently the strength of the system is basically the strength of the gel. This interpretation is further strengthened by the analysis of the failure criteria. The strength of the mass in the dense state depends very largely on the strength of the soil; thus the confining pressure will greatly increase the strength, due to the imposed restraint forces. The difference between soils A, B, C, D and E is a gradation effect which will be analyzed later.

The effect of strain rate on the unconfined compression strength of the stabilized soil is indicated in Fig. 5. The slowest rate of the equipment available for use in this program was 0.025 in. per min. Within the strain rates tested, the maximum mean variation was less than 4 psi. This variation is about half of a standard deviation from the mean or within the 40 per cent confidence interval. On this basis it can be clearly established that for strain rates greater than 0.025 in. per min there is no effect of strain rate on unconfined compression strength. Much soil testing is done at a strain rate of 0.02 in. per min and slower. Many cohesive soils show a decided reduction in strength at lower speeds. The trend in this direction is indicated in Fig. 5. The equipment limitations for this program were such that no further interpretations can be made.

It is reasonable to assume that the confined compression tests will exhibit strain rate effects similar to those obtained for the unconfined state. The strain rate effect, if present, is an internal phenomenon and the imposition of external conditions, while possibly affecting the magnitudes of internal responses, will not modify the basic character of this response.

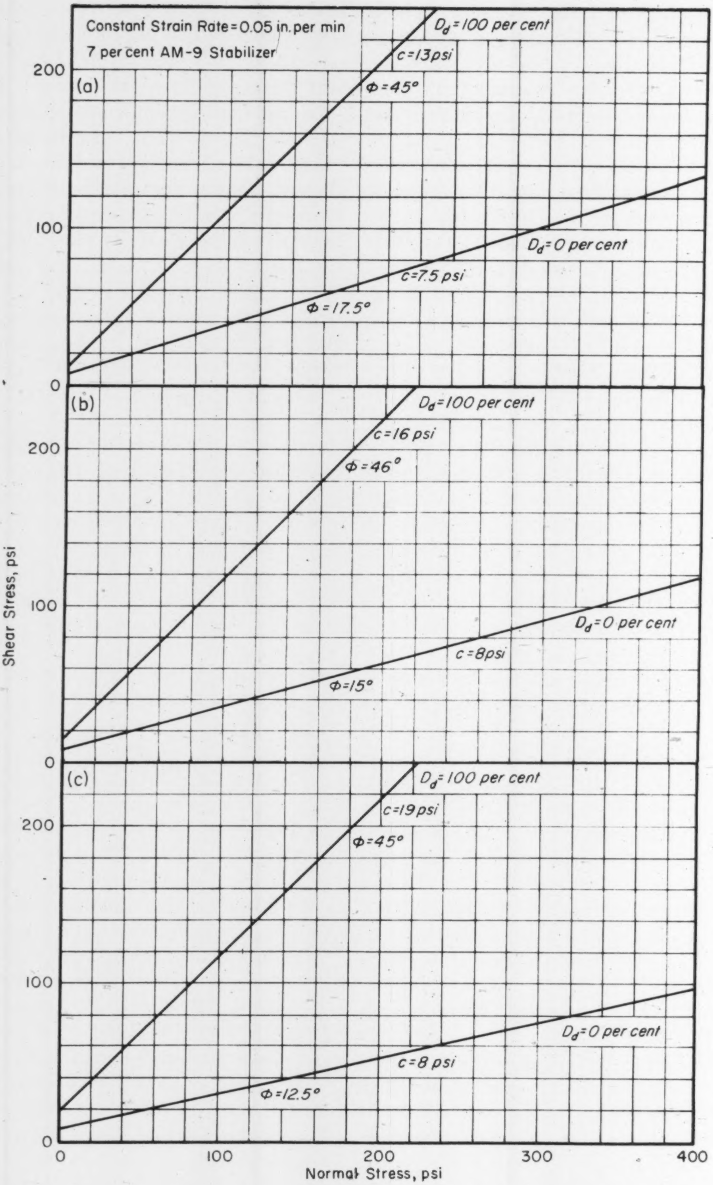
The hypothesis on the failure criteria was that failure followed the Mohr-Coulomb criteria. These criteria are presented in Figs. 6(*a*) to (*e*). These figures are based on the construction of the envelope of the three failure circles tested. As noted, the failure envelope is linear, thus implying that the Mohr-Coulomb criteria are satisfied. The failure criteria depart in magnitude from the well-established criteria in two distinct areas. In the first instance, the angle of internal friction for the stabilized soils in the loose state is decreased from that of the unstabilized state within a range of from 28 to 34 deg to a range of 8.5 to 24 deg. Excluding soil E, the upper limit of the range of the angle of internal friction was 17.5 deg. This is a substantial reduction in the internal friction of the system of stabilized soil as against the unstabilized state. The decreasing influence of the soil on behavior is again apparent. The fact that soil E departs from this criterion in magnitude only mitigates the factor of effect, and supplies an additional factor —gradation. This latter will be interpreted later. In the dense state, the angle of internal friction is that of the parent soil alone, or departs so little from it as to be inconsequential. We may thus conclude that the gel investigated does not affect the internal friction of the system in the dense state.

Another change in behavior due to the addition of stabilizer is the introduction of internal tension (cohesion) into the system. While the magnitude of this internal tension is small, never exceeding 1.5 ton per sq ft and as low as 0.5 ton per sq ft, it has a decided influence in strengthening the system.

The difference in failure behavior in the loose and dense states is shown in Fig. 7.

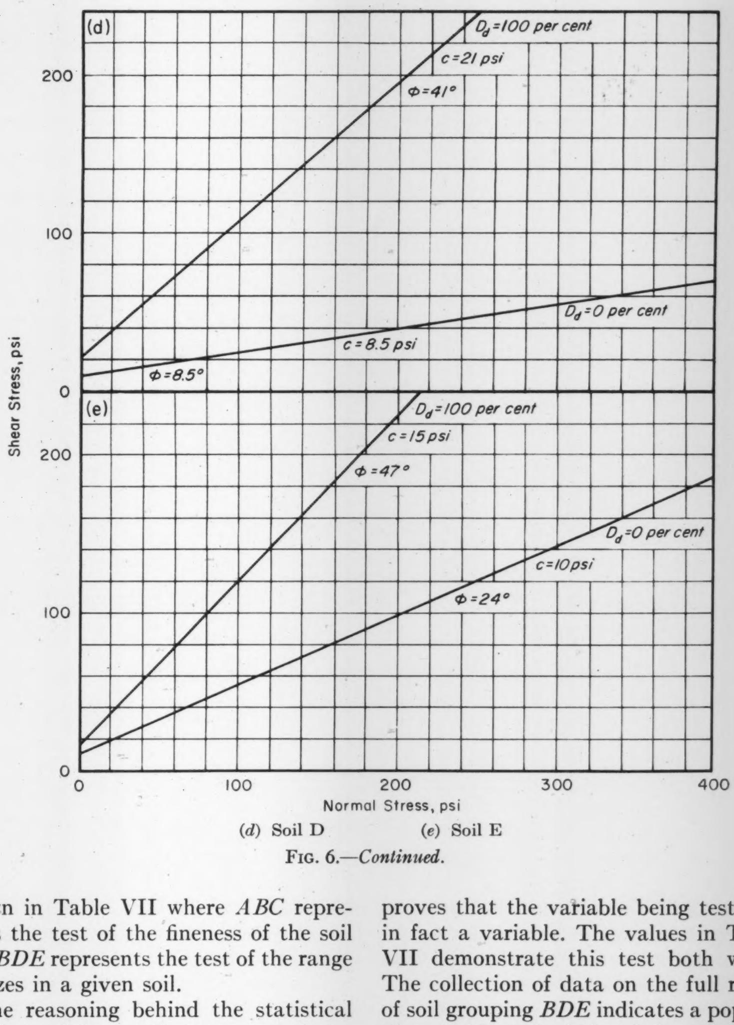
The dense state is characterized by an intergranular fracture at a definite angle. The loose state failure is more complex, starting with a split due to the Poisson effect and then developing to a failure along a plane of shear. In the dense state the gel content is at a minimum and the mechanism of failure is frictional, grain-to-grain. The loose state, however, contains more voids filled with gel, and the failure is more a failure of the gel than in the dense state. Extrapolating to the gel independent of the soil, the failure should be a split along the weakest system of crosslinks.

The validity of the hypothesis in relation to the effect of soil properties and strength was checked in accordance with a statistical analysis of groups for soils with common characteristics. The hypothesis of this investigation stated that the mean grain size and the range of grain sizes were a significant factor in the strength of the stabilized soil. Five soils were used to test its validity, three of which were of different mean grain size while three differed in range of grain sizes. The tests were performed in the unconfined strength condition on the assumption that a similar interpretation would be true for the confined strength condition. The statistical analysis is



(*a*) Soil A (*b*) Soil B (*c*) Soil C

FIG. 6.—Mohr Coulomb Failure Envelope.



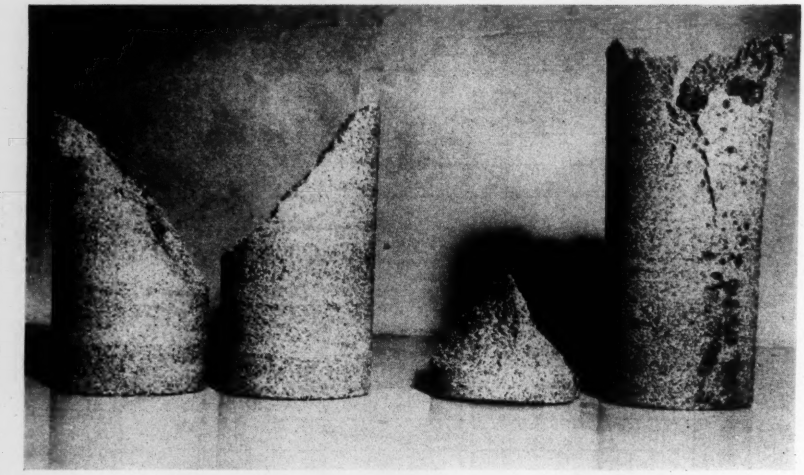
(*d*) Soil D (*e*) Soil E

FIG. 6.—*Continued.*

shown in Table VII where *ABC* represents the test of the fineness of the soil and *BDE* represents the test of the range of sizes in a given soil.

The reasoning behind the statistical test is as follows: The measure of significance of a single variable tested is reflected in the dispersion of the data taken over this variable. Thus if the dispersion as measured by the standard deviation is small, the test fails and the variable being tested has in fact no significance. On the contrary, if the standard deviation is large, then the test proves that the variable being tested is in fact a variable. The values in Table VII demonstrate this test both ways. The collection of data on the full range of soil grouping *BDE* indicates a population standard deviation below that for the same soils tested individually. This indicates that for the soils tested there is no influence on strength accruing from range of grain sizes.

In order to determine the influence in the loose and in the dense states of range of sizes, the data were broken down to these relative density states and ana-



(*a*) Dense (*b*) Loose

FIG. 7.—Failure of Stabilized Soil.

lyzed independently. The results taken on face value would confirm the previous interpretation of the independence of the range of grain size as a measure of strength. The specimen size for this analysis was quite small, and the results of soils B and D biased the results of soil E. An examination of Fig. 3 and Fig. 6(*e*) indicates that at 0 per cent relative density soil E showed significantly different strengths and failure conditions than all the other soils. This would indicate that the soil gradation has an influence but only beyond a definite lower limit. Thus the gradation does have an effect on the strength, but only for the better graded soils. The better graded soils will insure grain-to-grain contacts, and thus the strengths of the stabilized mass will be partially due to the soil and partially due to the gel. For the gradations measured at 100 per cent relative density, the analysis indicates that the effect of gradation is small, at best.

TABLE VII.—STATISTICAL REGRESSION ANALYSIS OF UNCONFINED COMPRESSION STRENGTH IN TERMS OF SOIL GROUPS.

Soil Groups	S_E , psi	Relative Density D_d , per cent	Number of Test Samples, *n*
ABCDE.....	9.3	0 to 100	56
ABC.........	11.5	0 to 100	33
BDE.........	4.4	0 to 100	34
ABC.........	2.68	0 to 36	13
ABC.........	15.96	94 to 100	6
BDE.........	3.85	0 to 36	12
BDE.........	3.83	91 to 100	9

The effect of fineness of the soil can be ascertained by means of the same statistical test. In the loose state, the low value of the standard deviation indicates that the strength is independent of the fineness. The reason for this is that the manner of failure in the loose state is through the gel. The large deviation in the dense states indicates that the wetted surface is of predominant importance and that the fineness is a highly significant feature of the strength criteria.

In general, the relative magnitudes of the deviations for the fineness measurement and the gradation indicate that the fineness is the more significant feature of the strength. This interpretation should be approached with caution, however, since the more poorly graded soils tend to bias the statistics.

With regard to the relation of the soil properties *versus* the strength at 100 per cent relative density, the results for unstabilized soil are somewhat different than would be expected. The strength increases as the fineness, D_{50}, decreases, all other variables remaining constant. The soil itself exhibits a certain strength, due predominantly to the frictional resistance preventing grain slip. The addition of the gel introduces internal tensile stresses, binding the grains together and raising the restraint level. This results from the gel filling the voids.

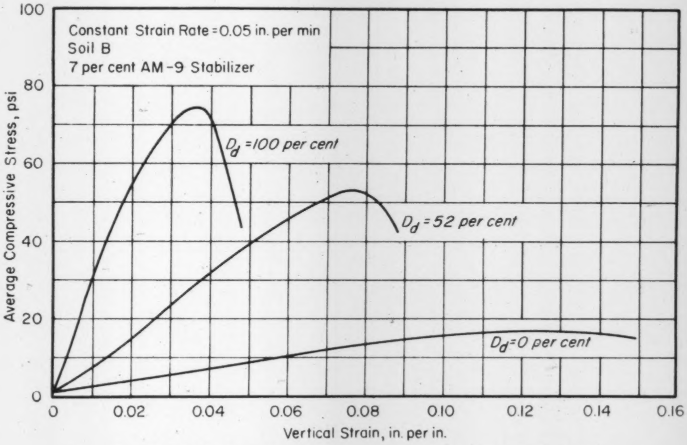


FIG. 8.—Typical Unconfined Compression Stress-Strain Behavior.

The mechanical behavior of soil initially stabilized with an adhesion chemical will be increased several fold.

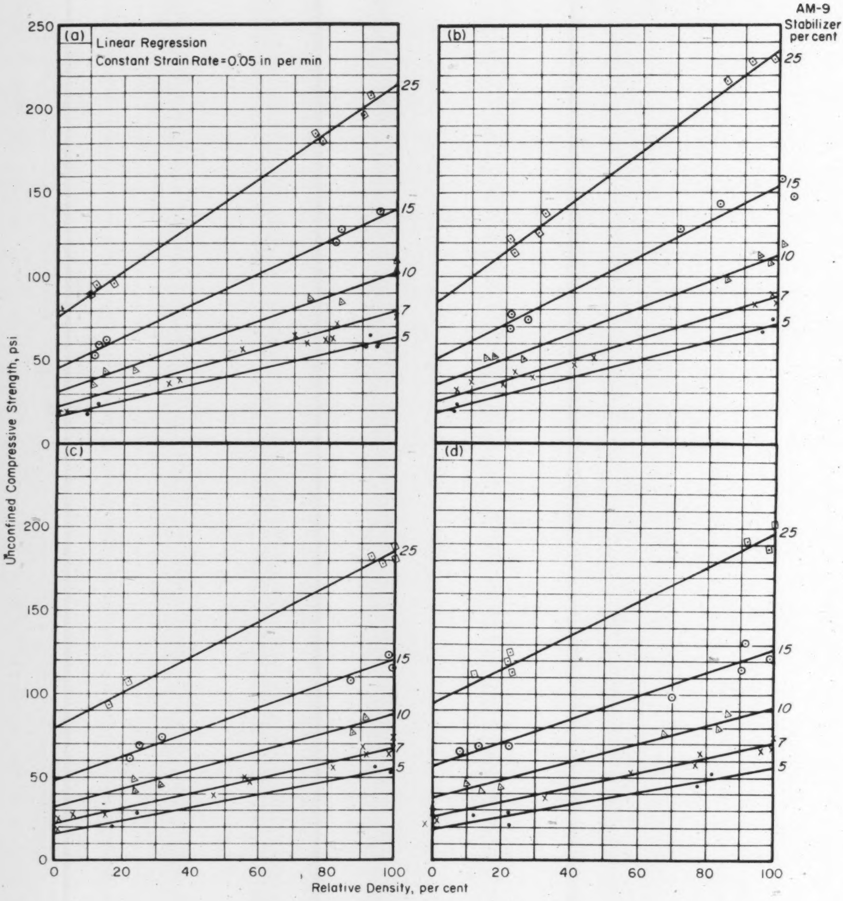
The internal tensions for a fully saturated newly gelled soil emanate from two actions: (*a*) the chemical adhesion between chemical and soil particle, (*b*) the tensile bond forces within the polymer itself.

The magnitude of the internal tensions is dependent on the relative density of the soils. The denser a given soil the closer the grains. The closer the particles the shorter and stronger will be the chemical bonding links between grains.

The fineness of the soil will influence the relative magnitudes of the adhesive bonding links. The soil fineness will be inversely proportional to the amount of adhesive surface available and thus inversely proportional to the internal tension available.

The effects of the gradation on the internal tension forces are governed by the coarser fractions. The greater the percentage of the coarser fractions the smaller the available adhesive grain surface and the lower the internal tension.

Examination of the stress-strain curves of the five stabilized soils at various relative densities indicates that the vertical strain at the maximum load



(*a*) Soil B
(*b*) Soil C

(*c*) Soil D
(*d*) Soil E

FIG. 9.—Effect of Monomer Concentration.

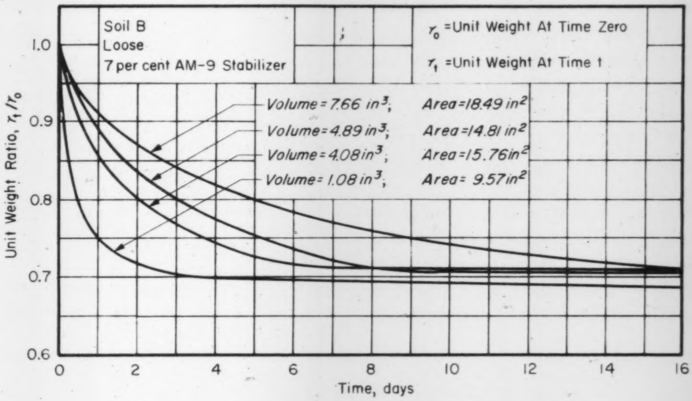


FIG. 10.—Unit Weight Decay as a Size Function.

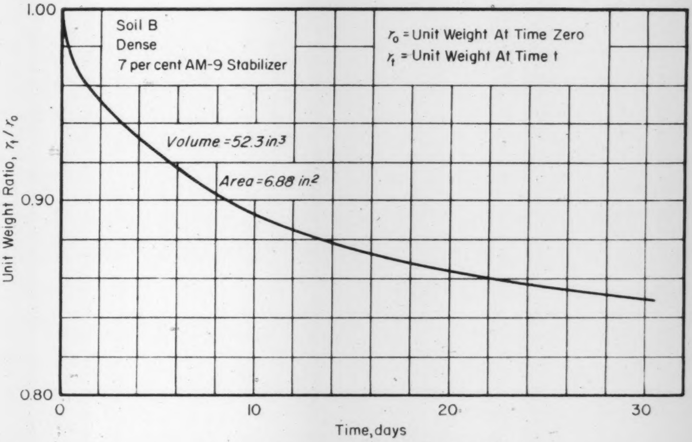


FIG. 11.—Typical Unit Weight Decay.

increases with a decrease in relative densities. A typical curve is presented in Fig. 8. In the dense state the grain compliance is such that there will be small deformations prior to slip and maximum load. In the loose state, the particles not being as closely together, more slip will occur before failure and thus more deformation.

The effect of confining pressure increases the strength and the vertical strain at maximum stress. This is simply the mechanical effect due to the external boundary conditions strengthening the entire system.

The general stress-strain relations are nonlinear and time dependent. The initial section of the curve shows a higher degree of linearity than the sections at relatively high strain. In this early stage the internal tensions tend to hold the grains in contact, producing a particle deformation, predominately elastic. If there was not full grain contact prior to testing, the initial movement serves to bring the grains into contact; this accounts for the initial concavity of the stress-strain curve. Immediately prior to failure, the grains incur friction slip, resulting in nonrecoverable strain. The effect of the friction slip on the stress-strain properties is more pronounced in the dense state due to the greater particle compliance. This effect is shown by large permanent deformations resulting from a load-unload cycle at a small percentage of the maximum strain. No creep tests were performed, but it can be assumed that the stabilized soil will undergo a creep deformation.

Stabilizer Concentration:

A series of unconfined compression tests were run on soils B, C, D, and E at varying concentration of the stabilizer. The intent of this series of tests was to determine quantitative information on the change in strength with changes in chemical content. For concentrations other than 7 per cent Stabilizer AM-9, tests were run only on the loose and dense states. (Since the previous test series proved the linearity between relative density and strength the same form of relation was considered to hold true for all chemical content.)

These test results are the results of 117 independent usable tests (11). A linear regression analysis was performed for each test series, establishing a least square mean line for the relation between relative density and unconfined compression strength. These regression lines are presented in Figs. 9(*a*) to (*d*), along with the data points.

Effects of Drying:

The effects of aging were tested in two ways: (*a*) by air drying a series of cylinders of varying size and measuring the time dependency of volume and unit weight change and (*b*) a series of identical sized cylinders were permitted to age and were tested in unconfined compression at various intervals. The change with time in the unit weight of the specimens is shown in Figs. 10 and 11.

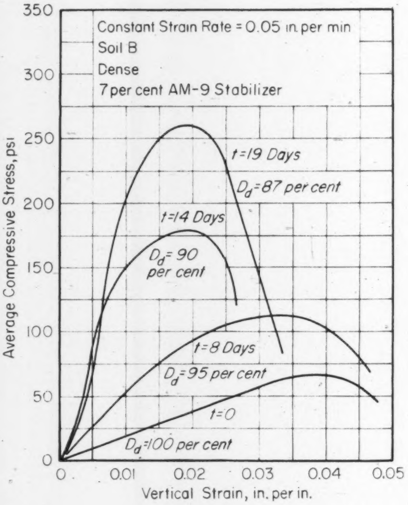


FIG. 12.—Unconfined Compression Stress-Strain Behavior During Air Drying.

The results of the tests show that there was no measurable volume change during the aging period through the point of equilibrium.

The unconfined compression strength tests of the soils indicated that after a few days the strength decreased with relation to the freshly gelled state, but beyond this point proceeded to increase until, at the end of 35 days, the unconfined compression strength was in the order of 10 times the original strength of the stabilized soil. A series of typical stress-strain curves during the aging process is shown in Fig. 12.

As drying progressed another phenomenon was observed. The outer surface of the specimen hardened and the inner core remained in its original softer state. The thickness of this outer hardened ring increased as the drying progressed until it comprised the total specimen at the end of the drying period.

The drying phenomenon starts at the outer boundary surface where macroscopic extensional cracks form in the gel. As these cracks form, they permit new surfaces of evaporation to develop and the gel tends to shrink toward the particles. The particles themselves are bound at the contacts by gel which is relatively unexposed; thus, the particles will remain in their original glued position. This explains the constancy of volume. Since the gel is mostly free water which is evaporating, the total unit weight of the specimen will be reduced. As the air voids in the outer ring increase, the rate of drying in the region will increase. In addition, the presence of these air voids permits the extension of the drying process to the inner elements of the mass. The net effect of this action is to achieve a negatively exponential drying curve. Due to the greater number of free surfaces, the evaporation process will proceed at a faster rate in the outer ring than inward. Thus, the outer boundary surface will probably have reached a state of equilibrium while the inner portions are still in the very early stages of drying.

As the gel shrinks toward the soil grains, a capillary meniscus is formed which will greatly magnify the strength of the soil. A microphotograph of the stabilized soil at the end of the shrinkage process is shown in Fig. 13.

FIG. 13.—Stabilized Soil After Air Drying.

The rate of increase of unconfined compressive strength is a function of the strain mechanism and the rate of drying. The material during any phase of the process of drying is a two-phase material, consisting of a hardened outer shell and a soft core. These two materials are joined in such a manner as to require complete continuity of radial and tangential stresses and vertical and radial displacements along the cylindrical boundary between materials. The test conditions require that the entire system be under a uniform displacement.

The increase in strength parallels the increase in the hardened outer core and the development of capillary stresses.

A typical failure is shown in Fig. 14. The character of the failure appears to be a modified cup-cone failure with the hard outer portion failing in shear along an oblique slip plane while the soft inner portion has an almost horizontal failure surface. This phenomenon partially follows the failure of sandstone cylinders observed by von Kármán (7). von Kármán found that failure of brittle materials in compression began at the cylindrical surface and was concentrated along conical surfaces which formed a wedge into the middle portion of the test specimen. von Kármán's test material was generally uniform, and the material here tested was not. Thus, the stress conditions must be modified to account for the geometry of the material.

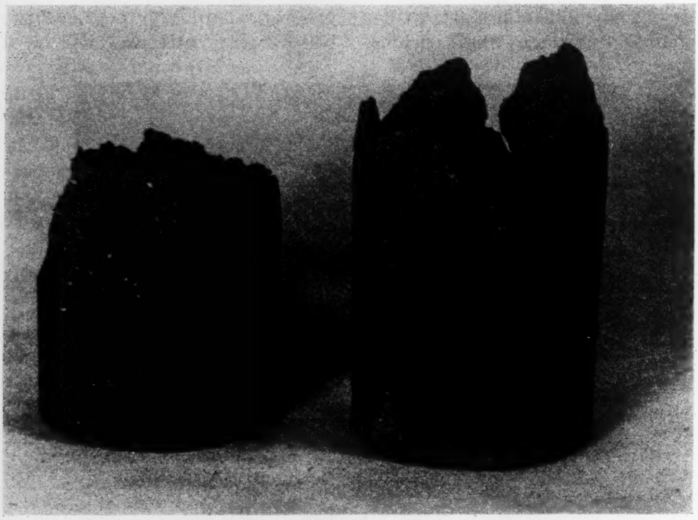


FIG. 14.—Two-Phase Failure.

Alternate Wetting and Drying:

In order to determine the effect of cycles of alternate wetting and drying, a factorial experiment was established on soil B in the dense state. The specimens were molded for a relative density between 87 and 100 per cent. The factorial experiment was designed to determine two effects: (1) the effect on strength of the number of cycles of wetting and drying, and (2) the effect on strength of the length of the cycle of drying or wetting.

The samples were cast, dried, and then soaked for the requisite times listed in Table I. The measure of response was the unconfined compression strength tests which were made after the last soaking cycle. Test results are presented in Fig. 15. An analysis of variance and F-testing of the test results indicate the following:

(*a*) The effect on strength of alternate

	Drying Period, days						
Number of Drying and Wetting Cycles	0	1	3	5	10	15	30
1	1.00	0.92	0.74	0.58	0.59	0.53	0.51
	1.00	0.85	0.74	0.62	0.51	0.50	0.54
	1.00	0.86	0.76	0.63	0.47	0.50	0.52
2	1.00	0.79	0.69	0.57	0.56	0.53	0.45
	1.00	0.77	0.67	0.60	0.50	0.56	0.45
	1.00	0.63	0.67	0.60	0.53	0.57	0.50
3	1.00	0.77	0.45	0.45	0.57	0.57	0.47
	1.00	0.73	0.58	0.49	0.59	0.56	0.45
	1.00	0.75	0.49	0.45	0.57	0.56	0.45

FIG. 15.—Dry-Wet Unconfined Compressive Strength Response.

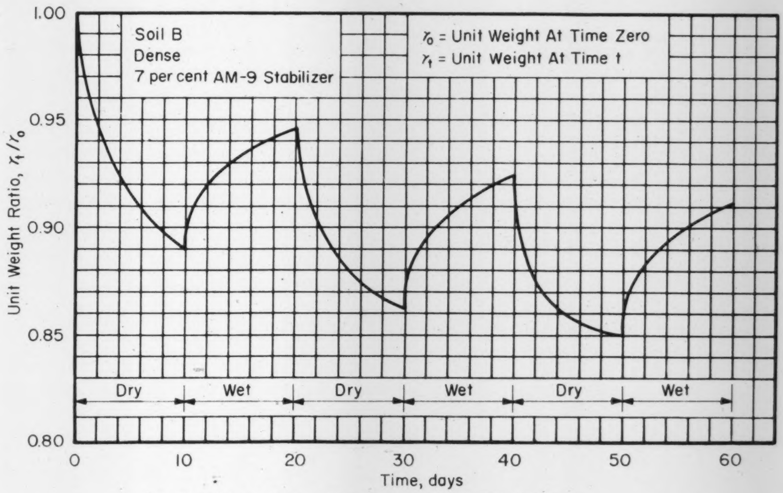


FIG. 16.—Typical Dry-Wet Unit Weight Variation.

cycles of wetting and drying was of no significance for drying periods of 10 days and longer; (*b*) The effect of alternate cycles of wetting and drying had no influence on strength. The only factor influencing strength reduction was the total number of drying days. Thus, 3 drying days at 3 cycles or a total of 9 drying days gave a strength reduction equivalent to that attained after 10 drying days at one cycle; and (*c*) The mean equilibrium strength after drying and wetting was 52 per cent of the original strength. The total range of deviations from the mean is 8 per cent on either side of the mean.

Thus, in the measured time range, a single influence only is to consider—the length of drying time up to the equilibrium condition of maximum strength decrease.

When the specimen dries, the chemical bonds are stretched and broken. If the effect on chemical bonding only is considered, since no capillary stresses are effective in the rewet state, the specimen will become potentially weaker as the drying process lengthens. Thus, the polymer will have lost much of its strength on rewetting when the capillary stresses are relieved. It is logical therefore that the rewet strength should be inversely proportional to the length of drying time up to a point where complete equilibrium is achieved in the extensional rupture of chemical bonds. When this point of equilibrium is attained, all the bonds likely to rupture will have broken and the effect of cycles of wetting and drying will be nil.

The typical total unit weight change curves of Fig. 16 indicate a single general trend, namely, that the unit weight at the end of the drying cycle is progressively reduced as the number of previous cycles increases. This reduction is greater than the increase in unit weight due to the wetting cycle, and indicates that after the initial drying the range of fluctuation of unit weight is increasing. This weight reduction is the measure of the free water held in the gel and the soil voids after the initial drying. After the first drying period the wetting fills the voids formed and a relatively small amount of water is absorbed by the gel. The subsequent drying drives out the void water and along with it more of the gel water than had been previously evaporated. A subsequent rewetting fills the voids anew and again increases the weight.

CONCLUSIONS

Many of the conclusions which may be drawn from this study apply in some degree to the mechanisms of behavior of granular soils when treated with any adhesive chemical:

1. The strength of the stabilized soil in the loose state is fundamentally governed by the gel and only slightly modified by the strength of the soil.
2. The strength of the stabilized soil in the dense state is fundamentally governed by the soil and only slightly modified by the strength of the gel.
3. The stabilizer will introduce internal tensions in the stabilized mass, the magnitude of which will depend on the chemical nature of the stabilizer.
4. The angle of internal friction in the dense state for stabilized soil is of the same magnitude as for the unstabilized mass.
5. The angle of internal friction in the loose state of a stabilized mass due to grain lubrication and weakness of gel as a void filler is markedly reduced from its unstabilized value.
6. The Mohr-Coulomb failure criterion is valid for the stabilized mass.
7. In the loose state, the strength of

the stabilized mass is independent of the fineness of the soil.

8. In the dense state, the strength of the stabilized mass is dependent on and increases with the fineness of the soil.

9. In the loose state, the strength of the stabilized mass will be modified by the gradation for size ranges greater than a limiting value. The better the gradation the greater the strength.

10. In the dense state, the strength of the stabilized soil will be relatively independent of the gradation.

11. The fineness of the soil is relatively more important than the gradation in terms of the strength of the stabilized soil.

12. There is no significant effect of strain rate on the strength of the stabilized soil for strain rates greater than or equal to 0.025 in. per min.

13. There will be no noticeable volume change in fully saturated soils subject to axially symmetrical evaporation surfaces.

14. Drying of soil stabilized with Stabilizer AM-9 will produce an increase in strength which will be 10 or more times the freshly gelled strength.

15. The increase in strength due to drying will be due to the imposition of capillary forces.

16. Drying of stabilized soil will proceed in layers with the rate of drying varying between outer and inner layers, the outer layers drying at the fastest rate.

17. The rate of drying will be dependent on the surface area available to evaporation at a given instant and also on the vapor pressure differential across a free boundary. The rate of drying is inversely proportional to the surface area available.

18. Drying follows an exponential decay law in which the volume and surface area influence the frequency of the exponential.

19. Failure by straining of partially dried stabilized soil will initiate at small strains in the dry portion and propagate to the wet area of the soil as modified by the constraining effects of the dry area.

20. Extended time of drying and cycles of wetting and drying will reduce the wet strength between 44 and 60 per cent of the strength of the freshly stabilized soil for a minimum drying length of one day for soils in the dense state.

21. There is no real effect from several alternate cycles of alternate wetting and drying. The effect of strength reduction is due solely to the total number of days of drying. The specimens tested indicated a maximum strength loss after a total of 9 drying days.

22. Successive wetting-and-drying cycles result in a net unit weight reduction at the end of the drying period.

Acknowledgments:

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MEASUREMENT OF SOIL PRESSURE ON A LATERALLY LOADED PILE*

By Hudson Matlock[1] and E. A. Ripperger[1]

Synopsis

Direct measurement of soil reaction against a laterally loaded pile is impractical with the instrumentation presently available. It is possible, however, to determine the pressure indirectly by measuring strains in the pile and converting these strains to bending moments from which the pressure is then obtained by double differentiation. To obtain satisfactory accuracy in the pressure determinations, a high degree of precision in the moment measurements is required. The order of accuracy required in the measurements, and special procedures for obtaining such accuracy are discussed A set of sample results is presented to illustrate the effectiveness of the special techniques and procedures described.

Methods presently available for design of laterally loaded piles in soft clays generally fall into one of two categories. Either it is assumed that the pile can be treated as a beam on a series of elastic springs, or rough approximations are made concerning the distribution of soil reactions. The development of either of these approaches, or of some alternate method, into a completely rational procedure based upon the results of laboratory or field soil tests will depend upon how successfully experimental data can be obtained. It is particularly important that reliable measurements of the soil resistance - pile displacement relationship for a wide variety of conditions be obtained.

The first steps toward a general solution of the problem of designing laterally loaded piles by making use of field determinations of soil resistance may be found in the work of McClelland and Focht (**1**)[2] and Mason and Bishop (**2**). Progress toward the final solution will depend in part upon how successfully further resistance-displacement data for pile-soil systems can be obtained.

Methods for Measurement

Several methods for making direct soil reaction measurements have been proposed and some have been tried, but mostly with indifferent success. Pressure gages distributed along the pile can measure only the reaction on the area of contact with the soil, whereas the desired quantity is the total reaction per unit length of pile. Consequently, even if pressures are measured directly, further analytical work, and possibly further assumptions, are required to obtain the reaction acting against the full width of the pile.

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[2] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 1259.

The difficulties associated with direct measurements may be avoided if the reaction distribution with respect to depth is determined indirectly from measured moments in the loaded pile. However, the moments must be measured with a high order of precision to make the calculated distribution of reaction reliable. The object of the discussion which follows is to show what order of accuracy is required in the measurements, to describe techniques which make the required accuracy possible, and finally to present data which show the extent to which these requirements can be met.

ANALYSIS OF PRECISION REQUIRED

The load p acting on an element of an elastic beam is related to the bending moment M by the familiar equation:

$$p = \frac{d^2M}{dx^2}$$

where x is the distance along the beam. Expressed in terms of finite differences, this becomes:

$$p_0 = \frac{1}{h^2}(M_{-1} - 2M_0 + M_{+1})$$

where h is the interval between points at which moments are measured, and the subscripts indicate the number of intervals from the midpoint at which the indicated pressure is found. The percentage error in the load due to a total error $\Delta\eta$, where

$$\eta = M_{-1} - 2M_0 + M_{+1},$$

is:

$$\frac{\Delta p}{p_0} = \frac{\Delta\eta}{\eta}$$

Clearly, in those regions where moments are changing almost in proportion to depth, the value of η and of the soil pressure is very small, and a small measurement error results in a large percentage of error in the calculated load.

To evaluate the order of precision required in the moment measurement, a hypothetical problem will be considered. Let it be assumed that a pile is driven in an ideally elastic soil in which the ratio, E_s, of soil reaction to pile deflection at any point is proportional to the depth below the surface. Stated mathematically, this is equivalent to $E_s = kx$. The data necessary for calculating the moment in the pile are assumed as follows:

$k = 0.015$ kips per sq in.[3]
length of pile = 600 in.
EI of pile = 11.66×10^6 kips per sq in.
lateral thrust applied at the mudline = 16.67 kips

Solution of the differential equation by finite differences, with a spacing of 6 in., gives the following set of moments in the vicinity of the maximum soil reaction (3).

Depth, x, in.	Moment, M, in.-kips
42	594.8
48	648.6
54	692.7
60	727.0
66	751.7

Assuming that these represent the correct moments, suppose that a positive error of 1 per cent (6.9 in.-kips) has been made in the determination of the moment at a depth of 54 in., but the moments at 42, 48, 60, and 66 in. are correct. At 54 in., then

$$\eta = 648.6 - 2 \times 692.7 + 727.0 = -9.8$$

$$\Delta\eta = 2 \times 6.9 = +13.8,$$

and

$$\frac{\Delta p}{p_0} = \frac{+13.8}{-9.8} = -1.4$$

Thus, an error of 1 per cent in one of the measured moments causes an error of -140 per cent in the reaction. Of course, if the error had been made in M_{-1} or M_{+1}, the effect on p_0 would have been only half as much, but opposite in sign. It should be noted, however, that the error in p_0 would still be $+70$ per cent, and the error in the pressure computed at the point where the moment error was made would be in the order of -140 per cent just as it was at the 54-in. depth.

If the spacing h is changed, and the accuracy of moment measurement remains unchanged, the errors in the calculated reactions are related as follows:

$$\frac{\Delta p_2}{p} = \left(\frac{h_1}{h_2}\right)^2 \left(\frac{\Delta p_1}{p}\right).$$

Thus, increasing the spacing by a factor of 2 results in a division of the error by a factor of 4 but at the expense of increasing the interval along the pile considered in each reaction computation. This means that the resolution with respect to depth is reduced.

To reduce the effect of experimental error, the measured values can be smoothed in various ways. Care must be exercised in this, however, lest *bona fide* variations in the measured moments be smoothed away. One method which has been used with satisfactory results consists of fitting a cubic polynomial by the method of least squares to successive sets of five equally spaced points and then differentiating the polynomial at the midpoint of each interval. This procedure results in the following general formula:

$$\left.\frac{d^2M}{dx^2}\right|_{x=x_0}$$

$$= \frac{1}{7h^2}\,[2M_{+2} - M_{+1} - 2M_0 - M_{-1} + 2M_{-2}]$$

From this result, it is seen that now

$$\eta = 2M_{+2} - M_{+1} - 2M_0 - M_{-1} + 2M_{-2}$$

This formula applied to the data in the example above gives:

$$\eta = -68.0$$

and

$$\frac{\Delta p}{p} = \frac{+13.8}{-68.0} = -0.20.$$

The error in the vicinity of maximum pressure is thus reduced from 140 per cent to 20 per cent. Part of this improvement is due to the smoothing and part is due to the increased interval considered.

Clearly, a high order of accuracy in the moment measurements is required if p is to be obtained from the measured moments, to a satisfactory degree of accuracy.

The feasibility of measuring moments with the necessary precision can be estimated best if all the possible sources of error are examined. Methods for reducing or eliminating errors from the various sources can then be considered.

The only feasible method available for measuring moment in a pile driven in the ground requires that the bending strains be measured using resistance strain gages. With the stiffness of the pile known, these strains can be converted to moments. Errors in the strain measurements can result from factors directly connected with the gages, such as,

1. Errors in gage positions and orientations,
2. Errors in gage sensitivity factors,
3. Creep and hysteresis in the gage bonding medium, and
4. Electrical leakage from gages to ground.

The instrumentation used to measure the change in strain-gage resistance may introduce errors through:

1. Lack of sensitivity,
2. Errors in calibration,
3. Faulty design,
4. Lack of stability, and

5. Personal errors in reading or operating the instruments.

With careful attention to the above sources, errors can be made very small, but accuracy is limited ultimately by inelasticity and hysteresis in the pile material itself.

If *EI* for the pile is used for the conversion of the strain to moment, any inaccuracy in the determination of *EI* will be directly reflected in the moment values. For determination of pressure, however, it is not necessary that *EI* be obtained. The pile can be directly calibrated for moment at each station in terms of observed strain. It follows, of course, that inaccuracies in the calibration contribute directly to the total error in the moment measurements. Constant errors will contribute to the error in the reaction without amplification, but errors which are not uniform from station to station may be amplified considerably in the reactions, as indicated in the hypothetical example considered above.

TECHNIQUES AND INSTRUMENTATION FOR ACCURATE DETERMINATION OF MOMENTS

The techniques described below have been used to measure with satisfactory precision the moments in an actual test pile. This instrumented pile was 12.75 in. in diameter and had an embedded length of 42 ft. An extra 10-ft length was bolted onto the top of the test section, partly to serve as storage space for electrical cables during installation of the pile (4).

Strain Gage Selection and Installation:

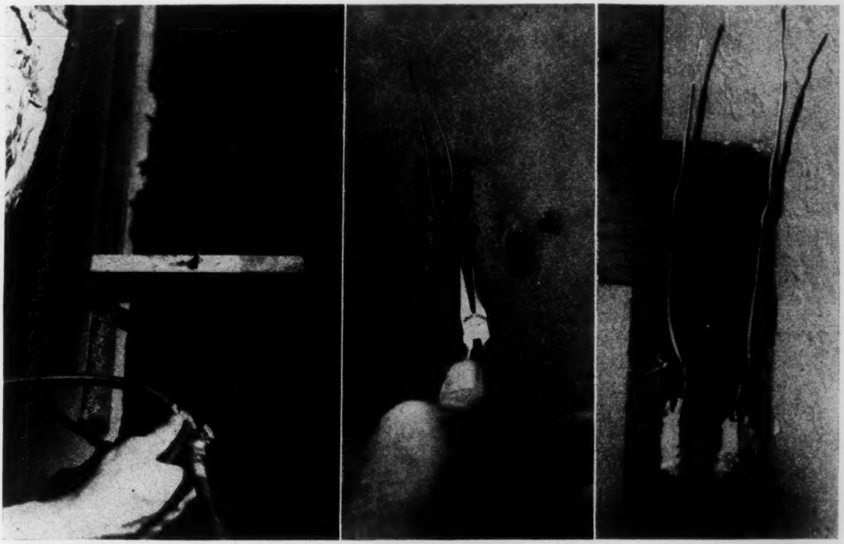
The only way to eliminate errors in longitudinal and lateral gage positions and in gage alignment is to use extreme care in installation. Convenient working conditions facilitate the use of extreme care. This convenience was provided in the test installation by cutting two 21-ft lengths of seamless pipe in half longitudinally and then mounting the four sections on blocks at convenient working height.

Errors in gage sensitivity factors can be circumvented for a test pile by calibrating the pile directly for moment as suggested above. The calibration technique used in the test program will be discussed later.

Creep and hysteresis in the gages can be minimized by proper selection of gage type and bonding medium. Baldwin SR-4 type ABD-1 gages were selected for this investigation because previous tests (5) had shown this particular type of gage to be exceptionally free from creep and hysteresis. The gage length of this type is $\frac{7}{8}$ in. and the nominal resistance is 350 ohms.

Before installation, the actual resistances of the gages to be used were measured to the nearest 0.01 ohm. The gages were then paired so that the difference in resistance between gages in each pair was no more than a few hundredths of an ohm. These were mounted in the pile so that the gages in each pair were diametrically opposed. This precaution insured bridge balances near the zero point of the instrument and eliminated the need for zero-balancing circuits which are likely to introduce instability. As a further precaution, the gages with the lower resistances were installed near the bottom of the pile and those with the larger resistances were placed near the top. This tended to compensate for differences in lead-wire resistances to the gages, and thus make the special circuitry used more effective in minimizing contact and lead-wire resistance changes. If a complete four-arm bridge had been installed at each point, the special circuitry would not have been required. However, considerable flexibility in measurement possibilities would have been lost. With the arrangement used, any gage could be compared to any other gage, or to any of the ten unstressed dummy gages installed in the pile. Thus, the loss of any one gage during driving and subsequent testing would not result in the complete loss of a station. A total of 70 active gages and 10 dummy gages was installed. This gave a total of 35 measuring stations, with a spacing that varied from 6 in. near the top to 48 in. in the lower portion of the pile.

Immediately prior to installing the gages, the interior of the pile was thoroughly sandblasted at the points where the gages were to be located, to insure a perfectly clean surface. Phenolic resin cement was used to apply the bakelite-backed gages. The only variation from recommended procedure for installation was the placement of a thin layer of glass fibers directly under the grid-to-lead junctions. Experience has shown that this precaution prevents the lead-wire junction from cutting through under the clamping pressure and coming into contact with the base metal. A special clamp, shown in Fig. 1(*a*), was used to apply, through an aluminum plate and Neoprene pad, the pressure needed to hold the gage, and a coiled spring in the clamping device maintained the proper pressure during curing. The other views in Fig. 1 show the attachment of very fine and flexible lead-wire extensions and securing of the junction with Armstrong type A-2 epoxy adhesive to prevent breakage during driving. The effectiveness of this method of strain relief in the lead wires is indicated by the fact that not a single connection was broken during service.



(*a*) Sandblasting of pile surface. (*b*) Connection of flexible leads. (*c*) Junctions secured with Epon.

FIG. 1.—Installation of Gages.

The heating necessary for curing the

phenolic resin used for bonding was done in the specially built curing oven, as illustrated in Fig. 2. This box was built of plywood and was lined with Fiberglas and aluminum foil insulation. Wire heating elements were suspended on porcelain insulators below the lids of the box. These lids may be seen at the left of the box in the figure. The temperature in the box was controlled by a thermostat. Uniformity of the pile temperature during curing was carefully checked using surface-type thermometers and Tempilstiks.

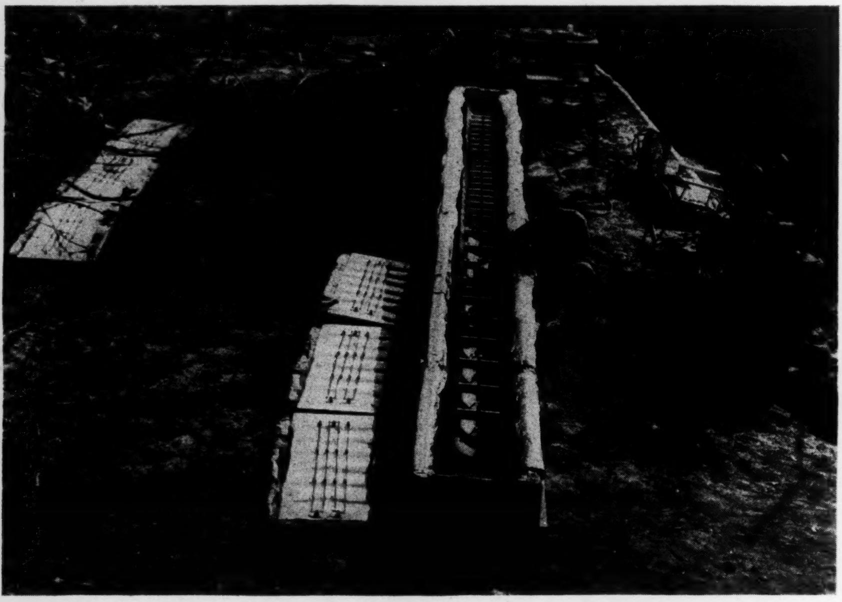


FIG. 2.—Electrically-Heated Box for Curing the Phenolic-Resin Strain-Gage Cement.

Waterproofing of Strain Gages:

Minimum resistances to ground of 50 to 100 megohms are usually recommended for ordinary strain-gage practice. Variations in a shunting resistance above this value usually do not cause significant changes in the strain gage bridge. However, where extremely good stability is required and ultra-sensitive instrumentation is used, the resistance to ground should be 500 megohms or more.

Several years of study of waterproofing by the authors has indicated that adequate protection of gages on driven foundation piling is extremely difficult to obtain. Where water is in immediate contact with a gage installation, the combined effects of water pressure and the shock of driving cause entry of small amounts of moisture. Resulting electrical leakages produce an effect similar to the shunting produced by a resistance connected in parallel with the gage.

Many methods for waterproofing gages are to be found described in the literature on stress analysis (**6–10**). However, most of these methods have not been proved to give absolute and permanent protection, particularly under the severe conditions where installations on driven foundation piling are exposed directly to water. For this reason, the test pile was provided with a closed end and was carefully leak-tested after the parts were reassembled and welded together. Since there still remained the possibilities of moisture reaching the gages due to damage during driving or from atmospheric humidity during testing, the gages were waterproofed by what appeared to the authors to be the best method available at the time.

Polyethylene, which is classed as a plastic but which chemically is a wax, is almost perfectly resistant to the passage of moisture. Furthermore, it is inert and therefore cannot affect paper and nitrocellulose gages by migration of plasticizers. It is a thermoplastic and can be molded and fused. Therefore, covers for the strain gages were made of polyethylene. The lead wires were also insulated with the same material, not only because of the waterproofing quality, but also to permit fusing of the lead wires into the covers. The point of lead-wire entry has been observed to be a critical point for moisture entry. Cables with extra-thick polyethylene insulation on each conductor were fabricated especially for this investigation. Extrusions of the same grade of polyethylene were obtained for blank stock for the gage covers to make fusing to the lead-wire insulation easier.

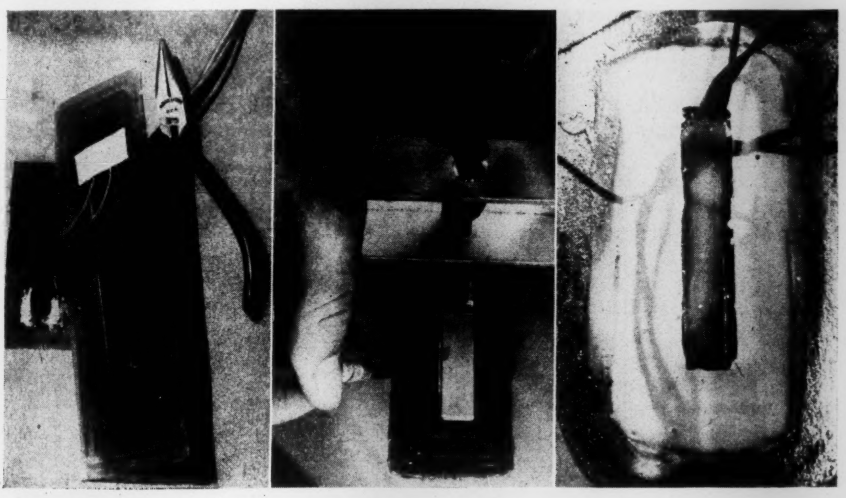

(*a*) Connections at the underside of the polyethylene covers.
(*b*) Cementing and coating of covers with adhesive.
(*c*) Covers secured with Epon.

FIG. 3.—Installation of Waterproofing.

The polyethylene covers were cemented in place over the strain gage installations with a special resin cement. This resin cement was the only one found which adhered readily to polyethylene; it had very poor mechanical strength and served primarily as a seal between the polyethylene covers and the pile surface.[3] Mechanical strength for

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[3] Since the completion of this investigation, some important advances have been made in the adhesives field which make it possible to cement polyethylene to itself and to other materials more effectively.

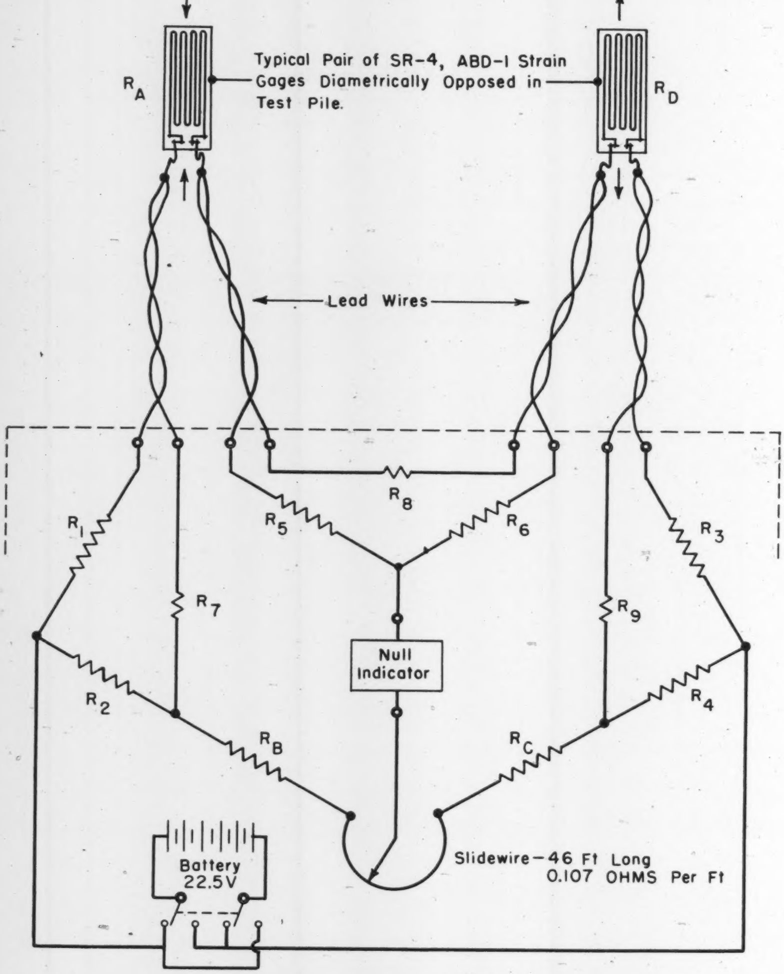
Typical Pair of SR-4, ABD-I Strain Gages Diametrically Opposed in Test Pile.
R_A
R_D
Lead Wires
R_1
R_7
R_5
R_8
R_6
R_3
R_9
Null Indicator
R_2
R_B
R_C
R_4
Battery 22.5V
Slidewire—46 Ft Long 0.107 OHMS Per Ft

FIG. 4.—Special Strain-Gage Bridge Circuit Used.

holding the covers in place was provided by a covering of epoxy adhesive.

A laboratory test of this waterproofing method was conducted prior to installation of any of the covers in the pile. In this test, a pair of gages was mounted on a steel strap and waterproofed by the technique described above. The strap was connected to a steel rod and submerged in a tank which could be sealed. The rod passed through a packing gland in the cover of the tank. With the cover in place, the tank was filled with water under a pressure of approximately 90 psi. To produce the effects of impact, the end of the protruding steel bar was struck with a hammer. The gages were left in the test tank for approximately ten days and subjected to impacts periodically. Satisfactory performance under these conditions indicated that this method of waterproofing would be satisfactory for the field installation. Waterproofing of all the gages in the test pile was then completed in accordance with this technique.

No water entered the test pile during driving; consequently, this waterproofing was not given a severe field test. Resistance to ground of several of the gages did drop below the 500-megohm level several months after the pile was driven, but these resistances were brought back up to a satisfactory level by putting silica gel in the pile. This drop was an indication, however, that the waterproofing was not completely successful. Close-up views of the polyethylene covers and the details of installation are shown in Fig. 3.

Instruments and Circuits for Strain Measurements:

The precision required in the moment data for successful determination of soil pressures by double differentiation dictated the use of manually operated instruments as opposed to automatic recording instruments. Direct-current circuits were selected to secure maximum dependability and stability.

The normal procedure planned for reading the gages was to connect them in pairs in two adjacent arms of a Wheatstone bridge circuit. However, in the event any gage should be lost during handling and driving, the remaining gage would be read by comparing it with one of the ten dummy gages in the pile.

In order to minimize errors in reading which might be caused by changes in lead-wire resistance and in contact resistances and to obtain strain readings of the highest precision, four lead wires were brought out from each gage. These lead wires were then connected into a circuit which minimizes errors arising as a consequence of long lead wires and contact-resistance changes (5). The general arrangement of the gages, lead wires, and the principal features of the bridge circuit are shown in Fig. 4. All of the circuit below the dotted line in this figure, with the exception of the electronic null indicator, is in an instrument case which houses the slide wire. It can be shown that if the resistances R_1 and R_2, R_3 and R_4, are properly chosen, changes in lead-wire or contact resistance will not result in any appreciable change of the bridge balance point. Virtually no current flows in the lead wires whose resistances are indicated as R_7 and R_9, and thus they act essentially as potential detecting leads.

The fixed resistances in the bridge circuit were provided by selected pairs of ABD-1 gages mounted on steel straps, $\frac{1}{4}$-in. thick, supported in a vertical position inside the case which housed the slide wire. The slide wire, which was especially designed to give the accuracy of measurement required in this investigation, is a piece of Manganin wire, 46 ft long, with a resistance of 0.107 ohms per ft. It is wound into a helical groove cut in the surface of a Plexiglas drum 6 in. in diameter and 11 in. long.

The slide-wire drum was calibrated by inserting 500-ohm resistors (accuracy, ± 0.05 per cent) in those arms of the bridge which in normal service the measuring gages would occupy. Each of the resistors was then shunted in turn with precalculated values of resistance set on a decade resistor having an accuracy of $\pm(0.05$ per cent $+0.30$ ohm). The precalculated values of decade setting were determined to give equal increments of change in resistance per unit of original resistance, $\Delta R/R$, in one bridge arm, equal to 100×10^{-6} ohms per ohm, which corresponded to about 2 in. in length along the wire. The entire usable length of the slide wire was then marked off in these increments. After marking the midpoints between the calibration marks, a corresponding 50-unit interpolating scale was placed on the sliding contact arm. The least division on the interpolating scale was 5×10^{-6} ohms per ohm. This allowed estimation to 1×10^{-6} ohms per ohm.

It is interesting to note that variations of as much as 2×10^{-6} ohms per ohm occurred in the 1-in. distance between 50-unit marks. Careful checking indicates that this represents variations in resistance per unit length of Manganin wire. Wire manufacturers have confirmed that this is about equal to the limit of manufacturing tolerance.

A Brown Electronik null indicator was used for indicating the bridge balance point. This instrument has an input resistance of at least 1000 ohms and a rated sensitivity of at least 1×10^{-9} amp per mm of deflection.

Estimating that the maximum reading error will be $\pm 2 \times 10^{-6}$ ohms per ohm, with two arms active and with a gage factor ($\Delta R/R$ per unit of strain) of 2.03, the maximum error in a single reading of strain in the pile is equivalent of $\pm 0.5 \times 10^{-6}$ in. per in. strain. It was observed that with stable load and temperature conditions in the pile, the sum of four readings, which constituted one observation at each station and was expressed in "slide-wire units," could be duplicated regularly within four slide-wire units (0.25×10^{-6} in. per in. of strain in either measuring gage) and frequently to within one or two units.

A standard reference for checking the instrument is provided by two ABD-1 gages mounted side by side on one piece of steel inside the instrument box. This reference pair can be switched into the bridge at any time in place of externally connected measuring gages.

Connections to the four lead wires from each of the gages were made through banana jacks mounted in aluminum terminal boxes. This system was used in preference to switching because of its simplicity, flexibility, and reliability. The cables and terminal boxes were stored within the head section of the pile during driving.

The procedure followed in taking readings contributed to the accuracy of strain determination. In addition, it had a number of self-checking features which made it possible to detect gross errors the instant an observation at a given station was completed.

After the first balance of the bridge was made, the reading was recorded on the tape of a printing-type adding machine. The battery polarity was then reversed and the bridge rebalanced. This reading was tabulated on the adding machine tape and added to the first reading. Clock time was printed on the tape also at this point for use later in interpolation required because of creep. The positions of the gages in the bridge arms were then reversed, the bridge was again balanced twice and the readings entered in the machine. The four bridge readings were then summed and printed on the tape. This sum represents four times the true instrument balance-point value. Any appreciable change in this quantity with different sets of gages is an indication of error somewhere in the observation. To obtain the indicated moment in slide-wire units, the sum of the first two readings is subtracted twice from the sum of all four readings. This gives $(Z_3 + Z_4) - (Z_1 + Z_2)$ which is four times the change in $\Delta R/R$. The net change in the indicated moment is obtained by subtracting the original no-load value from the indicated moment reading. Each step in the procedure and its significance are illustrated in Fig. 5.

This procedure not only makes higher reading accuracy possible by cancelling out the effects of thermal emf's, largely responsible for the differences between Z_1 and Z_2 and between Z_3 and Z_4 but also provides an immediate check for mistakes in reading and recording. Mistakes also can be detected and corrected satisfactorily long after the readings are made.

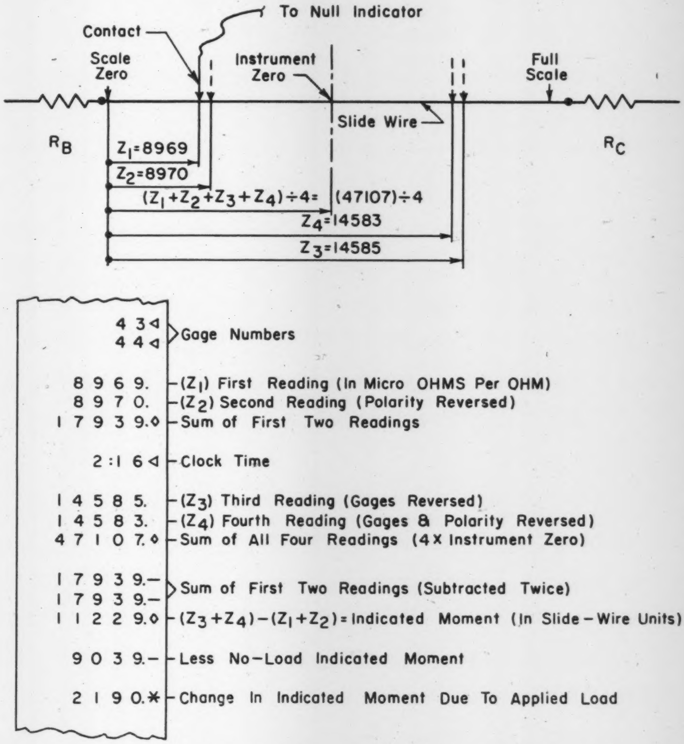
To Null Indicator
Contact
Scale Zero
Instrument Zero
Full Scale
Slide Wire
R_B
R_C
Z₁=8969
Z₂=8970
(Z₁+Z₂+Z₃+Z₄)÷4= (47107)÷4
Z₄=14583
Z₃=14585
4 3
4 4
Gage Numbers
8 9 6 9.
8 9 7 0.
1 7 9 3 9.
(Z₁) First Reading (In Micro OHMS Per OHM)
(Z₂) Second Reading (Polarity Reversed)
Sum of First Two Readings
2 : 1 6
Clock Time
1 4 5 8 5.
1 4 5 8 3.
4 7 1 0 7.
(Z₃) Third Reading (Gages Reversed)
(Z₄) Fourth Reading (Gages & Polarity Reversed)
Sum of All Four Readings (4 X Instrument Zero)
1 7 9 3 9.–
1 7 9 3 9.–
Sum of First Two Readings (Subtracted Twice)
1 1 2 2 9.
(Z₃+Z₄)–(Z₁+Z₂) = Indicated Moment (In Slide–Wire Units)
9 0 3 9.–
Less No–Load Indicated Moment
2 1 9 0.*
Change In Indicated Moment Due To Applied Load

FIG. 5.—Method of Recording Strain-Gage Readings.

Calibration of the Pile:

Since neither the cross-section of a pile nor the modulus of elasticity are precisely known, accuracy in the calculation of moments from measured strain requires that the pile be calibrated by applying known moments and measuring the corresponding strains at each station. It should be noted that if the pile is calibrated with a moment distribution exactly similar in form to that expected in the testing program, errors in the placement of the gages can also be nullified. It is usually not practical to calibrate with such a moment distribution, however.

The average stiffness of the pile, *EI*, is not required for moment calculations if the pile is calibrated, but it is needed if deflections are to be computed. It can be obtained when the pile is being calibrated.

The scheme which proved to be most satisfactory for calibration in the experimental work utilized the dead weight of the pile to produce a precise and stable change in moment. To obtain initial readings, the pile was first supported as a simple beam with one support at the flange and another at 480 in. from the flange or 24 in. from the pile tip. In this position, the reaction at the support near the pile tip was measured with an SR-4 load cell. The pile was then supported in a cantilevered position with a tie-down secured at the top end of the 10-ft head section, as indicated by the small sketches in Fig. 6. The change in moment could then be accurately computed at every gage station of the pile, and it was found that strain readings could be accurately reproduced. The change in bending stress near the flange was about 12,000 psi.

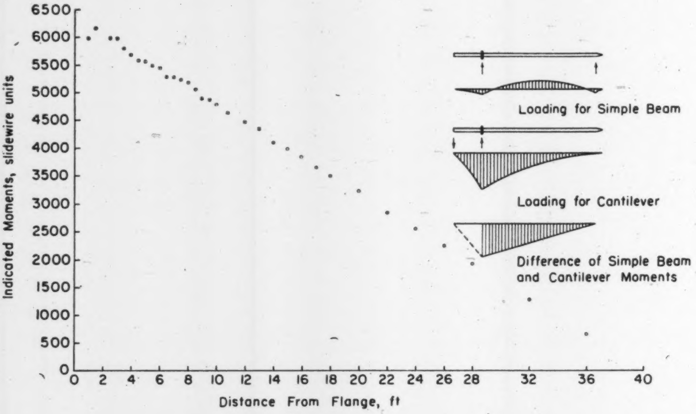

FIG. 6.—Pile Calibration Method and Results.

Calibrations were obtained under both positive and negative moment with respect to the pile. After one set of simple beam and cantilever observations were made, the pile was rotated 180 deg about its axis, as determined by a spirit level. All observations were then repeated.

Although most of the calibration work was done during cloudy and rainy weather, or at night, transient temperature changes were found to be sufficient to prevent complete calibration of all stations with one loading change. The elapsed time between cantilever and simple beam loadings was reduced to about 30 min by repeatedly alternating the support condition, with only a few stations being read before and after each change.

The results of the calibration for each station are plotted in Fig. 6. Each point represents the average of two independent determinations with the pile rotated 180 deg. The average variation between determinations for the two positions was 0.06 per cent. The variations from a straight line of some of the points in Fig. 6 are in the order of 2 or 3 per cent. This indicates the order of the error which would be introduced in the measurements of moments by the use of a calculated section stiffness.

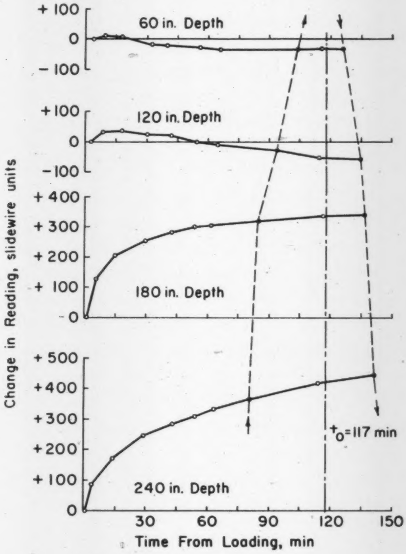

FIG. 7.—Creep Observations.

In order that the moments, and therefore the pressures, which may be induced in the pile by the driving can be determined, absolute-zero moment readings should be made before the pile is driven. An attempt to do this by averaging two sets of simple beam readings, one taken before and the other after a 180-deg rotation, was abandoned when it was found that transient temperature differentials between the two sides of the pile reduced considerably the relative accuracy of the zero readings which were obtained. A temperature differential of 1 F between the two sides of the pile would cause an error in indicated moment of roughly 60 slide-wire units. To obtain better values, it would have been necessary to thoroughly insulate the entire pile. The zero readings which were used in data reduction were determined after the pile was driven.

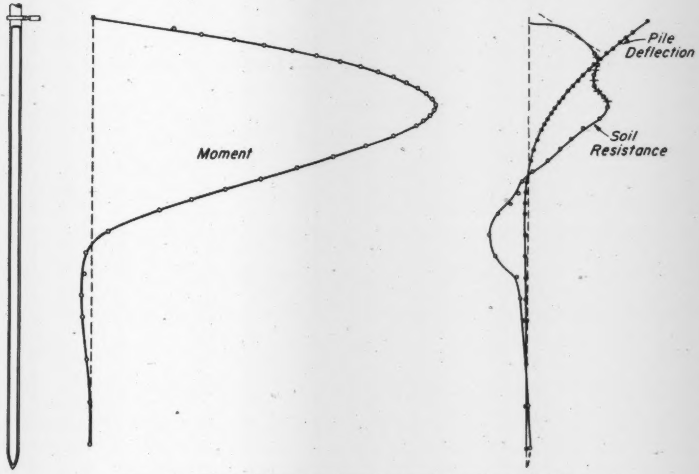

FIG. 8.—Typical Results from Lateral Loading of Pile.

INTERPOLATION FOR CREEP EFFECTS

No matter how accurately the strain at a given station and at a given time may be determined, it is clear that calculated pressures based on these strains may be practically worthless if the strains are changing rapidly with time and considerable time elapses between readings. To read the strain at 35 stations on the test pile following the procedure described above required at least 35 min. During this time, the strain in the pile was changing due to the effects of consolidation and shear creep in the soil. The procedure which was adopted for minimizing the error introduced by this sort of behavior required two complete sets of strain readings at each load. Also, starting immediately after loading, creep was observed at several selected stations until changes with time became sufficiently slow and linear to allow satisfactory interpolations to be made between two complete sets of readings.

The creep interpolation procedure is illustrated by Fig. 7. Observations taken during regular reading of all gages are shown with solid black circles in this figure. The connecting dashed lines with arrows show the sequence of reading up and down the pile. In processing the data, the value at time t_0 was obtained by linear interpolation between the two observations. By studying those stations which had been observed for creep, it was determined that the interpolation error was in nearly all cases negligible.

TYPICAL RESULTS

The test pile was loaded in increments with a controlled force applied at the mudline. A typical set of bending moments in the pile, determined by scrupulous adherence to the techniques and procedures described above, is shown in Fig. 8.

The deflection of the pile was obtained by numerically integrating the moment data and correcting for a slight amount of rigid body rotation.

Soil reaction on the pile as shown in Fig. 8 was obtained by least-squares fitting of a cubic polynomial to successive sets of five moment values as explained previously. In the zone of the 6-in. spacings near the top of the pile, two independent sets of calculations were made using alternate sets of gages at 12-in. spacing. These are indicated by the different symbols used for the plotted points on the reaction curve. Since each gage station was independently calibrated, these two alternate sets of pressure values are essentially independent of each other, and the agreement in this zone indicates the degree of reliability of the results.

It should be understood that by using a cubic polynomial the soil resistance values are based on the assumption that the pressure distribution is a straight line in the vicinity of the five points considered in each calculation. For the 12-in. spacing, this means that the pressure distribution is approximated by the best straight line fit over a 4-ft interval. It is possible to increase the resolution of determinations of pressure distribution by decreasing the spacing between stations. However, an increase in random scatter should be expected.

Expressions were also derived for off-center differentiations of the fitted polynomial for use at the top and bottom of the pile. Such differentiations result in an accuracy substantially less than that achieved by differentiation at the center of the interval. Near the top of the pile, the resulting straight dashed line at the top of the pressure curve in Fig. 8 is only a rough approximation of the true pressure distribution. Better results could have been obtained if this difficulty had been anticipated. Alteration of the loading connection could have provided for several additional gage stations above the mudline.

The general regularity of the plotted points in both the moment and the pressure curves is an indication of the consistency of results and therefore of the practicability of obtaining soil reactions by strain-gage measurements of bending strains.

CONCLUSIONS

Moment data of sufficient precision for calculating reliable soil reaction distributions on a laterally loaded pile can be obtained using the strain-gage method. However, careful attention must be given to producing the best possible data. Following are some of the considerations which are important:

1. Gages should be selected which exhibit a minimum of creep and hysteresis.
2. All leads and attachments must be adequately secured to prevent damage during driving.
3. A degree of moisture protection for the gages much better than usual practice is required.
4. Circuits and instruments must be designed for maximum stability and sensitivity.
5. Circuits which minimize the effects of lead-wire resistance changes should be used and techniques which eliminate the effects of contact potentials and resistances should be used.
6. The gage stations must be individually calibrated with precisely known moments applied to the pile.
7. Some smoothing should be applied to the data.

Acknowledgments:

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SPECIFIC GRAVITY OF MONTMORILLONITE CLAY CONSTITUENTS*

By J. E. Shea[1]

Synopsis

While running routine specific gravity tests on soils by air displacement, an unusually high value was obtained on a clay soil from Brown Field near San Diego, Calif. The presence of montmorillonite was suspected. A check of the geographic distribution of montmorillonite disclosed that the material is indigenous to the area. A carefully planned and conducted experiment then showed a definite increase in specific gravity of the material with decreasing moisture content when measured by air displacement. This phenomenon obviously could not be indicated by normal water displacement methods. An analysis of the material conducted by the Massachusetts Institute of Technology showed the material to contain a large percentage of montmorillonite. By a search of the literature definite information was uncovered showing that the "*C*" axis of spacing between the plates of montmorillonite varies with the moisture content and with the application of heat. The paper describes a simple method that, in the opinion of the author, can detect qualitatively the presence of montmorillonite clays in soils. It is believed that further research might develop a quantitative measure.

By use of a special method, engineers of the District Public Works Office, Eleventh Naval District have determined new sets of values for specific gravity of certain fine-grained soils. The research was accomplished in the Soil Mechanics and Paving Branch of the Eleventh Naval District in San Diego, Calif. under the general direction of the author by E. M. Lindahl, C. V. McElree, C. E. Beard, and W. M. Archibald.

The method involves the measurement of specific gravity by volume determination through the medium of gas displacement (in this case air) instead of the commonly used liquid displacement (water).

The air displacement instrument is called a volumeter.[2] Briefly, the instrument is composed of two chambers, one of which contains the sample. The air in the sample chamber is put under pressure to a fixed amount, after which it is allowed to expand into an auxiliary chamber with a resultant pressure drop.

The principle is based on the use of the gas laws which state:

$$\frac{P_1V_1}{T_1} = \frac{P_2V_2}{T_2} \quad \text{(1)}$$

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* Presented at the Sixty-first Annual Meeting of the Society, June 22–27, 1958.

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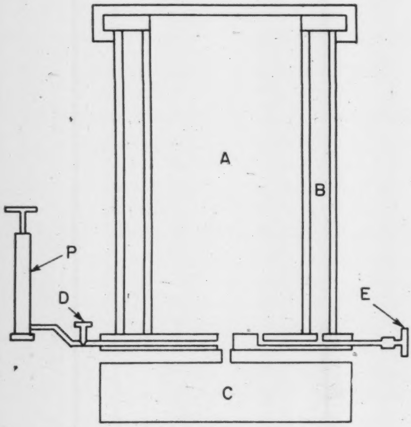
[2] U. S. Patent No. 2667782, *Engineering News Record*, May 4, 1950.

where P_1, and V_1 and T_1 are initial absolute pressure, volume, and temperature respectively and P_2, V_2 and T_2 are the final absolute pressure, volume, and temperature respectively after the connecting valve is opened.

Under the experimental conditions which prevail for the test, it may be assumed that there will be no change in temperature and therefore Eq 1 may be reduced to:

$$P_1, V_1 = P_2, V_2 \ldots\ldots\ldots\ldots (2)$$



A—Sample chamber
B—Auxiliary chamber
C—Pressure gage
D—Air intake
E—Valve connection between *A* and *B*
P—Air pump

FIG. 1.—Diagram of Volumeter.

Now, let P_b = barometric pressure, P_{g1} = initial gage pressure, P_{g2} = final gage pressure, A = volume of sample chamber, and B = volume of auxiliary chamber.

Then the left-hand side of Eq 2 above, representing initial condition before the connecting valve is opened, may be written:

$$(P_b + P_{g1})\,A + P_bB,$$

and the right-hand side of Eq 2 above, representing final condition after A and B are connected, may be written:

$$(P_{g2} + P_b)\,(A + B)$$

When the above terms are equated and expanded, we obtain:

$$P_bA + P_{g1}A + P_bB = P_{g2}(A + B) + P_bA + P_bB \ldots (3)$$

which reduces to:

$$P_{g1} = \frac{P_{g2}(A + B)}{A} \ldots\ldots\ldots (4)$$

From the above it may be seen that the volumes in the sample chamber are dependent only on gage pressure and are unaffected by either temperature or barometric conditions. Figure 1 is a diagram of the air displacement instrument.

In running a sample of the native clay materials from the Naval Auxiliary Air Station, Brown Field, Chula Vista, Calif., an unusually high specific gravity of over 4 was obtained by the method described above. All sources of possible error were checked, and as none could be found it was theorized that the pressurized air had penetrated voids in the clay particles that were inaccessible to water. The sample which showed the high specific gravity had been heated to approximately 400 F by inadvertence and had been tested in the volumeter shortly after cooling to ambient temperature. A check of the same sample with ASTM Method D 854 – 58[3] gave a normal specific gravity of 2.74. A microscopic examination was made of samples which were air-dried and heated to 212, 400, and 600 F and there were no discernible differences. Spectrographic analysis of the soils used were interesting but inconclusive due principally to the chemical similarity of kaolinite and montmorillonite.

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[3] Method of Test for Specific Gravity of Soils (D 854 – 58), 1958 Book of ASTM Standards, Part 4.

The hypothesis that air penetrated voids which water could not reach was investigated and discarded. It was noted, however, that the sample tested with the volumeter or by ASTM Method D 854 gave practically the same specific gravity under normal conditions of test.

It was then theorized that excessive heat caused a collapse of the particle structure for certain types of clays. A search of the literature disclosed the following facts:

(*a*) E. S. Dana, in his "Textbook of Mineralogy,"[4] states that montmorillonites are to be found in San Diego County (Brown Field) and that bentonite (principal constituent, montmorillonite) is found along the West Coast (Mojave). Kaolinite, on the other hand, is widely distributed (all other clays tested).

(*b*) According to recent investigations[5] there is a marked difference in the orientation and spacing between the plates which make up clay particles. Kaolinite clay plates are tightly held together whereas montmorillonite plates are disoriented and separated. This supports the belief that the Brown Field and Mojave clays contain important quantities of montmorillonites whereas other clays tested are composed chiefly of the kaolinites.

(*c*) According to John C. Hathaway, in "Studies of Some Vermiculite-Type Clay Minerals,"[6]

> "Heating the samples to 400 C causes a shift in the first-order peak to lower spacings without affecting the 7 Å or the 3.5 Å reflection which suggests that a kaolinitic mineral is responsible for most of the intensities of the 7 Å and 3.5 Å peaks. Samples 18-A, 675F, and 947 show similar effects, although a slight shift of the 14 Å reflection of 675F toward higher spacings suggests the presence of interstratified expanding layers. The 400 C heat treatment causes a collapse of the 14 Å spacing of samples 675F and 947 to much lower spacings than those obtained for the three remaining samples; the 7 Å spacings, however, survive relatively intact. The results of a step-by-step heating program on samples 1B are shown in .... A steady collapse of the 14 Å reflection with increasing temperatures occurs until a 10 Å spacing is reached at 800 C,"

and

(*d*) From "The Colloid Chemistry of the Silicate Minerals" by C. Edmund Marshall[7] the following is quoted.

> "This innermost interference, however, in the case of montmorillonite changed its position with water content, thereby clearly proving, since the other lines remained fixed, that it is caused by planes parallel to the cleavage, whose distance apart varied with the water content of the clay."

A check of the assumption concerning the presence of large amounts of montmorillonite clay was obtained through the courtesy of the Massachusetts Institute of Technology. The mineralogical analysis of a sample of Brown Field clay gave the following results:

Of the $-74\ \mu$ fraction:
  Hydrous mica clay, 85 per cent (equal proportions of illite and montmorillonite)
  Carbonate minerals, 13 per cent
  Organic matter, 1 per cent
  Free iron oxide, 0.8 per cent

In order to investigate this phenomonon of high specific gravity for montmorillonite clays a plan of test was developed to determine the causes for these unusual results. The testing method by air displacement had been used for over three years on hundreds of soil samples

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[4] E. S. Dana, "Textbook of Mineralogy," John Wiley & Sons, Inc. New York, N. Y. (1932).

[5] S. J. Gregg, "Surface Chemistry of Solids," Reinhold Publishing Corp., New York, N. Y. (1951).

[6] John C. Hathaway, "Studies of Some Vermiculite-Type Clay Minerals," *Clays and Clay Minerals*, Nat. Academy Science, Nat. Research Council, Publication No. 395, p. 76 (1954).

[7] C. E. Marshall, "The Colloid Chemistry of Silicate Minerals," Academic Press Inc., New York, N. Y., p. 7 (1949).

TABLE I.—DATA FROM TESTS ON BROWN FIELD CLAY.

Test Number	Wet Weight Sample, g	Weight Water, g	Weight Solids, g	Volume Sample, ml (Volumeter)	Volume Solids	Specific Gravity	
Brown Field Native Soil							
	Column 1	Column 2	Column 3 Columns 1 − 2	Column 4	Column 5 Columns 4 − 2	Columns 3/5	Remarks
1......	500	65	435	221.7	156.7	2.78	230 F—56 min
2......	500	55	445	216.7	161.2	2.78	230 F—91 min
3......	500	45	455	207.7	162.7	2.80	235 F—130 min
4......	500	35	465	198.2	163.2	2.85	235 F—170 min
5......	500	25	475	180.2	155.2	3.06	235 F—210 min
6......	450	15	435	143.2	128.2	3.39	235 F—245 min
7	450	10	440	134.2	124.2	3.55	+11 min at 400 F
8	450	5.5	444.5	115.2	109.7	4.05	+17 min at 400 F
9......	450	4.5	445.5	90.2	85.7	5.19	+10 min at 600 F
10......	460	16.0	444	156.7	140.7	3.16	Water added
11......	468	23.0	445	177.2	154.2	2.89	Water added
12......	483.5	38.5	445	198.2	159.7	2.79	Water added
13......	497	55	442	211.2	156.2	2.83	Water added
14	464.0	60	404	205.2	145.2	2.78	Water added
15......	468	67	401	207.7	140.7	2.85	Water added
16	482	82	400	223.2	141.2	2.83	Water added

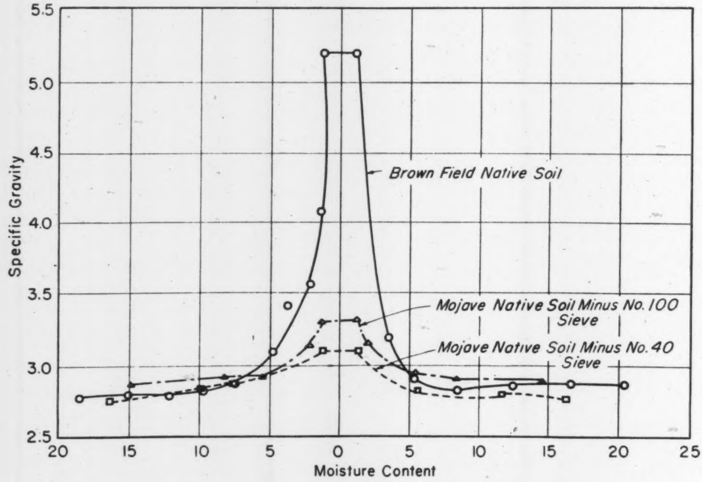

FIG. 2.—Specific Gravity Air Displacement.

and always gave results which could be checked, and occasionally were checked, by ASTM Method D 854 - 58. Also, the volumeter was used to determine successfully the specific gravity of portland cement and bentonite clays and to determine moisture content of wet samples and bituminous content of bituminous mixes. The data from the tests on Brown Field clay are given in Table I.

Of all of the soils tested, only two, Brown Field native material and the material from the Marine Corps Auxiliary Air Station, Mojave showed this difference in specific gravity to an important degree. Extensive tests were run on these samples and the results plotted (Fig. 2). The dry specific gravity of the material is plotted along the ordinate and the moisture content is plotted along the abscissa. The individual points were obtained by starting the test with a wet sample and determining the dry specific gravity as shown in Table II. The test data from the Mojave soil are not shown in order to conserve space.

It was never possible to test samples as dry as the heating made them, since during the process of testing they picked up approximately 1 per cent of moisture from the air. Therefore, the samples were gradually dried and the specific gravity measured at various moisture contents. After the sample was reduced to a minimum obtainable moisture content, water was added and a curve developed for specific gravity against increasing moisture contents. In all cases, the two curves are practically symmetrical. It is noted that after the material has been repeatedly dried and wetted the final specific gravities tended to increase slightly. A possible explanation is that some of the fine grains or crystals were permanently shattered.

Table III shows a very close check between the two methods for determining specific gravity of the soils containing montmorillonite when tested after normal drying and illustrates the important change that occurs in air displacement when higher heat is applied to montmorillonite clays.

Table IV summarizes the results obtained from the testing of several different materials and also gives certain other descriptive data pertaining to these materials for identification purposes.

Because the laboratory equipment used is primarily intended for engineering tests, it is not adequate to conduct delicate research work. It is evident that

TABLE II.—STEPS IN DETERMINATION OF DRY SPECIFIC GRAVITY.

Column 1 Wet Weight Sample	Column 2 $\frac{\text{Weight Loss}}{\text{Dry Weight}}$	Column 3 Dry Weight $\frac{\text{Sample}}{\frac{1}{100 + 2}}$	Column 4 Total Volume by Volumeter	Column 5 Volume Water Columns 1 − 3	Column 6 Volume Solids Columns 4 − 5	Column 7 Specific Gravity $\frac{\text{Column 3}}{\text{Column 6}}$
490.0 g	$\frac{50.7}{439.3}$ 11.54 H_2O per cent	$\frac{490.0}{111.54}$ 439.3 g	209.2 ml	490.0 −439.3 50.7 ml	209.2 −50.7 158.5 ml	$\frac{439.3}{158.5}$ 2.77

TABLE III.—SPECIFIC GRAVITY.

Material	ASTM Method D 854 - 58	Volumeter	Volumeter and Heat
Brown Field soil	2.74	2.77	5.19
Mojave soil (−No. 40 sieve)	2.74	2.75	3.08
Mojave soil (−No. 100 sieve)	2.75	2.76	3.31

there are many side effects and corollaries whose detailed investigation is suggested by this basic problem. A few obvious points are as follows:

(*a*) The effect (if any) of the residual pressure in the volumeter when readings are taken. This is the order of 5 psi gage pressure.

(*b*) Additional gas absorption on the particles when water vapor is replaced.

TABLE IV.—SUMMARY OF TEST RESULTS OF VARIOUS MATERIALS.

	Pass No. 200 Sieve, per cent	Silt, per cent	Clay, per cent	Liquid Limit	Plasticity Index	ASTM Method D 698 – 58 T,[a] Maximum Density Modified by Corps of Engrs.	Optimum Moisture, per cent	Specific Gravity, ASTM Method D 854 – 58	Specific Gravity Air Displacement (Wet Sample)	Specific Gravity Air Displacement (Wet Sample)	Remarks
Ottawa sand	0	0	0	non-plastic		..	..	2.65	2.67	2.67	
Portland cement	..	..	..	..	..	..	..	3.15	3.14	3.14	
El Centro native soil	98	51	38	56.0	30.0	116.3	14.7	2.70	2.83	2.87	
Camp Pendleton native soil	27	11	5	non-plastic		128.7	9.7	2.70	2.82	2.80	
Brown Field native soil	86	63	17	76.0	42.3	105.7	20.2	2.74	2.77	5.19	
Mojave native soil	30	24	0	33.7	13.2	123.6	12.0	2.66	2.76	3.00	Whole sample
Mojave native soil	45	36	0	..	..	..	..	2.74	2.75	3.08	–No. 40 sieve fraction only
Mojave native soil	73	58	0	..	..	..	..	2.75	2.76	3.31	–No. 100 sieve fraction only
Litchfield Park native soil	64	46	6	non-plastic		123.8	10.0	2.68	2.79	2.80	

[a] Tentative Method of Test for Moisture-Density Relations of Soils (D 698 – 58 T), 1958 Book of ASTM Standards, Part 4.

NOTE.—The slightly lower dry specific gravities obtained by water displacement are believed due to the presence of a variable amount of hygroscopic moisture at the start of the test.

(*c*) Affinity of the soil for water—that is, wetting ability.

(*d*) Surface area measurements, and

(*e*) Use of gases other than air to check results.

CONCLUSIONS

This paper discloses what is believed to be an entirely new soil measurement, and while practical application in engineering work is not apparent at this time, there is no doubt that further investigation and experience will disclose important uses. Based on the research conducted thus far, the following tentative conclusions seem to be warranted:

1. Apparatus used in these experiments would lend itself readily to such investigation.
2. The constituent particles of some soils are much heavier than previous measurements have disclosed.
3. Specific gravity measurements by presently practiced procedures are limited, and
4. The air displacement method provides a means of establishing qualitatively the presence of montmorillonite clays and further refinements will enable quantitative measurements.

ADDENDUM

Because the following is speculative and based on insufficient data it is not treated as a part of the paper. If 85 per cent of the material passing the No. 200 sieve is platey clay and this represents 86 per cent of the total sample of Brown Field soil then 73 per cent of the total sample causes the reaction described above and this equation may be written:

$$0.27 \times 2.77 + 0.73X = 5.19$$

and $X = 6.09$ which is the specific gravity of the collapsed particles of montmorillonite and illite.

To determine the per cent of montmorillonite in the total sample of Mojave soil the following is written:

$2.76\,(1 - X) + 6.09\,X = 3.00$ from which the montmorillonite is 7.2 per cent of the total sample.

Similarly the per cent of montmorillonite in the $-$No. 40 sieve fraction is 10.0 and in the $-$No. 100 sieve it is 16.7.

If, as would be expected, the activity is confined to the silt and clay fraction one would expect that in the case of the Mojave soil, the ratios of the per cents of montmorillonite would be equal to the ratios of silt-clay fraction thus:

$$\frac{\text{Total}}{-\text{No. }40}; \qquad \text{Silt-clay } \frac{24}{36} = 0.67;$$

$$\text{montmorillonite } \frac{7.2}{10.0} = 0.72$$

$$\frac{\text{Total}}{-\text{No. }100}; \qquad \text{silt-clay } \frac{24}{58} = 0.41;$$

$$\text{montmorillonite } \frac{7.2}{16.7} = 0.43$$

$$\frac{-\text{No. }40}{-\text{No. }100}; \qquad \text{silt-clay } \frac{36}{58} = 0.62;$$

$$\text{montmorillonite } \frac{10.0}{16.7} = 0.60$$

These ratios are interesting and suggest further experimentation to develop quantitative results.

DISCUSSION

Mr. T. W. Lambe.[1]—The author in his paper has reported very high specific gravity values measured by air displacement rather than the commonly used water displacement techniques. Such high values would indicate that air particles are entering parts of the clay aggregate which cannot be entered by water. This inference is not, however, correct. Water, being polar, is much more strongly attached than air to the soil mineral surface. The surface will preferentially adsorb moisture over air.

The very high measured specific gravities conflict with theoretical values computed on the basis of crystal structures. Such computations show that the specific gravity of the common clay-forming minerals should be in the neighborhood of 2½ to 3.

Mr. J. E. Shea (*author*).—The author agrees in general with Mr. Lambe. However, the explanation for the phenomenon is different from that suggested by Mr. Lambe. At one time the theory that air had penetrated voids inaccessible to water was considered, but was later discarded for the reasons suggested by Mr. Lambe. The explanation that is now proposed is that the particles of montmorillonite clay changed their structure under the influence of heat and that the plates which make up the crystals are brought closer together by a reduction of the "C" axis. Each particle has then reduced its volume with a consequent increase in specific gravity. Air is able to surround each of the particles and effectively measure this increased specific gravity.

A method of calculating molecular and formula weight from X-ray data can be found in a text on X-ray crystallography by C. W. Bunn, "Chemical Crystallography," Oxford University Press, pp. 185 and 186 (1945). If one assumes that the crystal structure remains unchanged and also assumes the molecular weight of the substance, it is possible to calculate an approximate specific gravity for normal conditions. However, this is a complete reversal of the research by the crystallographer whose purpose is to calculate molecular weight. On page 186 of the reference, the author, Bunn, describes a method of determining specific gravity by floatation and states further that the chief error in determining the molecular weight of a substance is likely to be in the value of the density. It appears evident that calculations of specific gravity of crystalline structures in their normal state are in no way related to the phenomenon described in the paper under discussion.

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[1] Professor, Massachusetts Institute of Technology, Department of Civil and Sanitary Engineering, Cambridge, Mass.

Proc. ASTM; Vol 58, 1958, pp. 1269-1277

THE COMPRESSIBILITY OF SANDS*

By J. E. Roberts[1] and J. M. de Souza[2]

The Soil Engineering Division of the Department of Civil and Sanitary Engineering at the Massachusetts Institute of Technology is conducting a research program aimed at a better understanding of the fundamental behavior of soils. One phase of this program is the study of the compression behavior of soils. The results presented in this paper were obtained from one-dimensional, high-pressure compression tests on soils. The research was originally concentrated on clays and shales, but the significant result of the investigation, to date, has been the discovery of the relatively high compressibility of quartz sands at high pressures due to shattering of individual grains. Compression indices from 0.5 to 0.7 have been obtained for quartz sand in the pressure range from 1000 to 10,000 psi.

Previously published work on sand compression which has been reviewed either has not shown the high compressibility (in part because the pressures used were not high enough) or has not recognized completely the significance and mechanism of the behavior. (A list of publications related to sand compression is given at the end of this paper.)

Apparatus and Procedure

Because of the high pressures involved, a special loading frame capable of applying constant loads as high as 50 tons for indefinite periods of time was designed and constructed. This equipment is shown in Figs. 1 and 2. It consists of a hydraulic ram (a cylinder and hydraulically operated piston) to apply the load and an emery-load cell to measure precisely the applied load. The pressure in the ram is maintained constant by an hydraulic accumulator which is merely a cylinder containing a bag which can be filled with nitrogen. The pressure developed in the nitrogen in the bag is transferred to the oil in the accumulator and ram. The gas pressure can be maintained constant by means of a reduction valve attached to a tank of compressed nitrogen.

A load increment can be applied almost instantaneously. If the gate valve (valve *B* in Fig. 2) is closed, pressure in the accumulator can be raised to the desired value without changing the pressure in the ram. After the oil pressure in the accumulator has been raised to the desired valve and the reduction valve set to maintain this pressure, the gate valve can be opened to increase the pressure in the ram. As the soil sample compresses under the increased load, the piston moves and oil flows from the accumulator to the ram. The reduction valve regulates the flow of gas from the nitrogen

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* Presented at the Sixty-first Annual Meeting of the Society, June 22–27, 1958.

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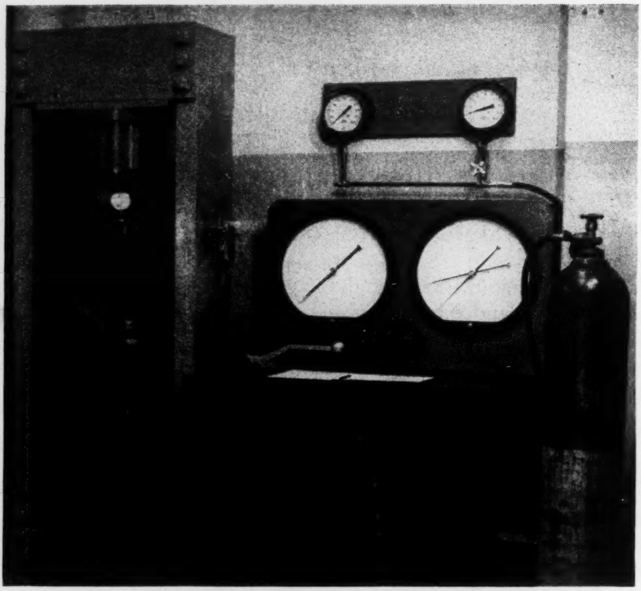

FIG. 1.—High-Pressure Consolidation Apparatus.

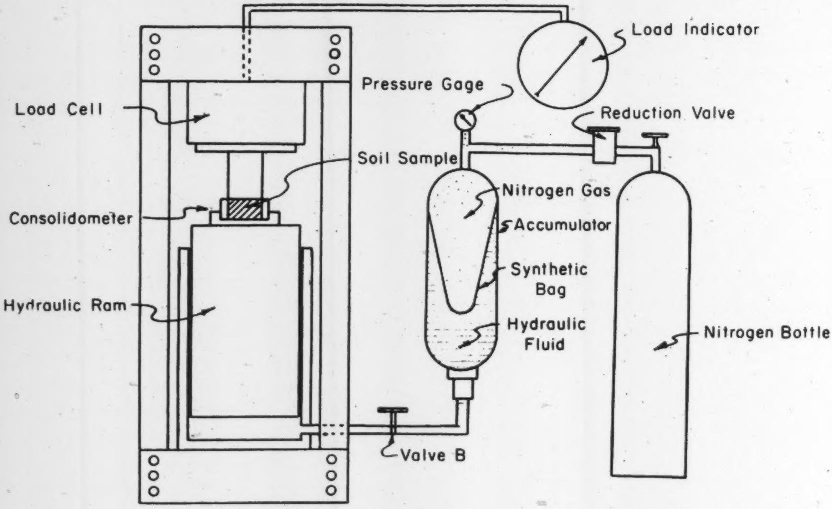
Load Indicator
Pressure Gage
Load Cell
Reduction Valve
Soil Sample
Nitrogen Gas
Consolidometer
Accumulator
Synthetic Bag
Hydraulic Ram
Hydraulic Fluid
Nitrogen Bottle
Valve B

FIG. 2.—High-Pressure Consolidation Apparatus (Schematic).

tank to maintain a constant pressure in the accumulator and ram.

The principal tests used in the investigation reported herein were one-dimensional compression tests on samples 1.13 and 2.75 in. in diameter and 0.35 to 0.75 in. in initial height.

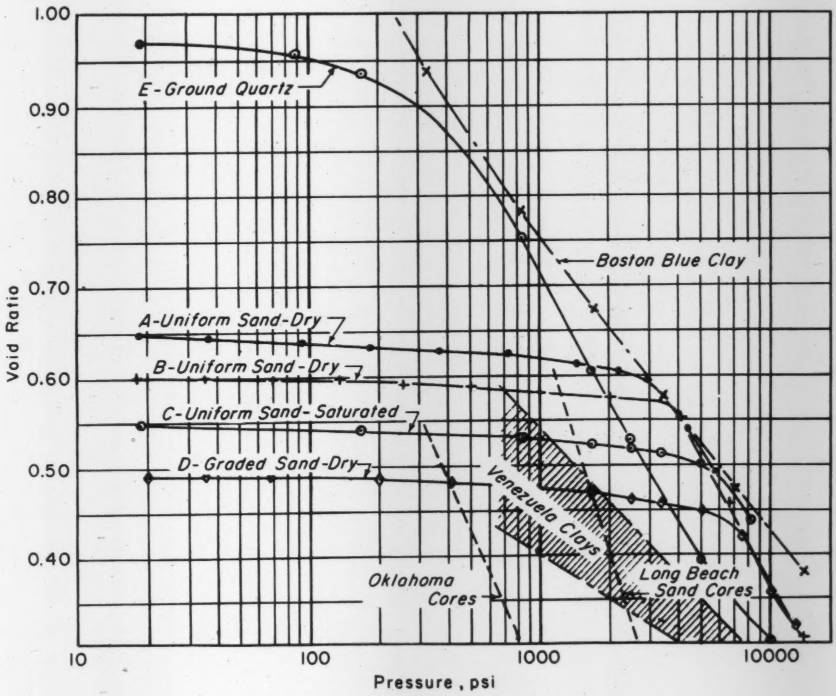

FIG. 3.—Compression Diagrams.

Tests on clays were conducted in a manner comparable to the normal consolidation test procedure, that is, a load increment ratio, $\frac{\Delta p}{p}$ of approximately unity was used and the sample was allowed to consolidate under the load increment for 24 hr before the next increment was applied.

Tests on the sand, in general, followed a similar procedure relative to load increments. However, the loads were applied gradually in order to avoid impact effects and the time allowed for compression was much shorter (in some tests 10-min durations were used). Generally, the same load-increment duration was used throughout a single test. Samples of dry sand were prepared for testing by pouring the dry sand into the consolidation ring and then jarring the ring slightly until a flat sand surface was obtained. Samples of saturated sand were prepared by allowing the sand grains to settle through water into the consolidation ring.

SOILS TESTED

Although the main purpose of this paper is to describe the experimental results of the compression tests on sands, the significance of the results can be better emphasized by comparing the compression curves obtained with those of certain clays.

One sample of a blue-gray clay (Boston blue clay) which is representative of the glacial clay found in the Boston Basin area was used. The sample tested has a liquid limit of approximately 50 per cent and a plastic limit of approximately 23 per cent. Based on the construction suggested by Casagrande (**7**)[3] this soil had a maximum past consolidation pressure approximately equal to 4 tons per sq ft.

Twenty samples of Venezuelan clays obtained from core samples taken from depths of from 2724 to 4769 ft below ground surface were also tested. These clay samples had liquid limits ranging from 27 to 67 per cent and plastic limits ranging from 15 to 26 per cent; when plotted on a plasticity chart the data fell in a band parallel to and slightly above the A-line.

Three tests were performed on a well-rounded uniform quartz sand known commercially as Ottawa sand. The uniform grain size was obtained by using only that part of the material which passed the No. 20 sieve (U. S. standard series) and was retained on the No. 40 sieve. The grain size distribution curve before testing is shown in Fig. 6(*b*); Fig. 7(*a*) is a microphotograph of the material before testing.

One test was performed on a well-rounded graded quartz sand having the initial grain size distribution shown in Fig. 6(*a*).

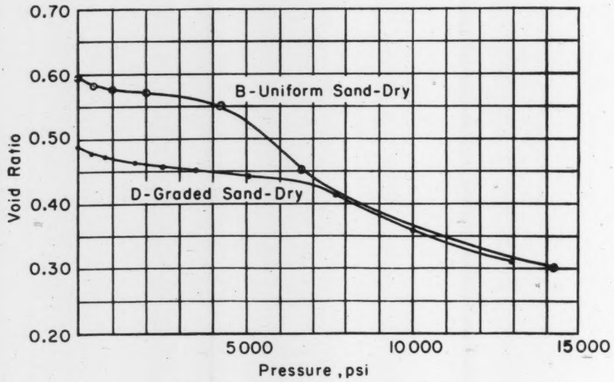

FIG. 4.—Compression Diagrams.

One test was performed on an extremely angular uniform quartz sand obtained by sieving ground quartz in the same manner as the well-rounded uniform sand.

TEST RESULTS

The results of the tests performed are presented in summary form in Figs. 3 to 6. Figure 3 presents the compression data plotted as void ratio *versus* pressure (log scale). The shaded area marked Venezuela clays represents the range of virgin compressibility obtained from the 20 samples tested. To obtain the shaded area, the virgin compression portion of the curve obtained in the test was extended as a straight line over the range shown even though in the actual test results most of the curves were straight over only some part of the range (many of the samples tested appeared to have been precompressed to pressures of the order of 2000 psi). The maximum compression index,[4] C_c, obtained on the Venezuela clays was 0.26. The sample of blue clay tested has a compression index equal to 0.33 over the same pressure range.

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[3] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 1276.

Also shown in Fig. 3 are two curves representing the variation of natural void ratio *versus* overburden pressure for sand cores taken from borings in California, and for shale cores from borings in Oklahoma (**21**).

The compression data obtained from tests *B* and *D* are replotted using an arithmetic scale in Fig. 4.

Figure 5 is a plot of the time curve obtained for the final load increment of compression test *A*.

Figure 6 shows the grain size distribution as a function of maximum applied pressure for the well-rounded uniform and graded materials.

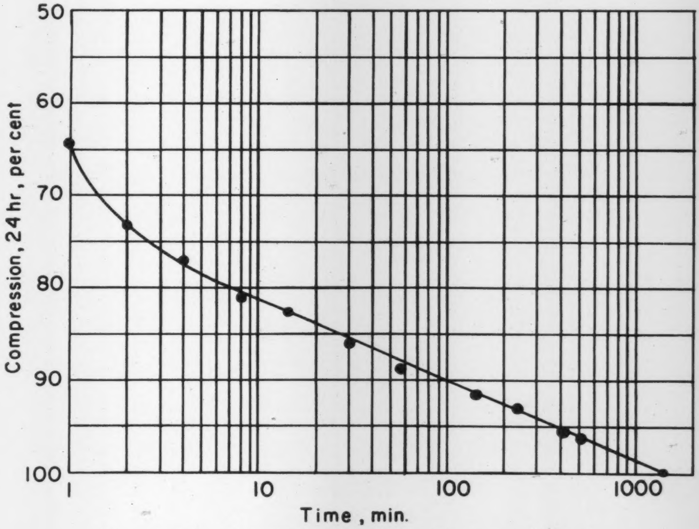

FIG. 5.—Time Curve for High-Pressure Increment on Sand.

Discussion of Results:

The most important fact brought to light by the test results is the high compressibility of sands at high pressures. At low pressures the sands are relatively incompressible.

At low pressures, the compression is due primarily to the shifting of particles by sliding and rolling. There is some elastic compression of grains and some crushing and breaking at the points of contact. However, Fig. 6(*b*) shows that pressures as high as 1000 psi cause only slight degradation on the uniform sand tested.

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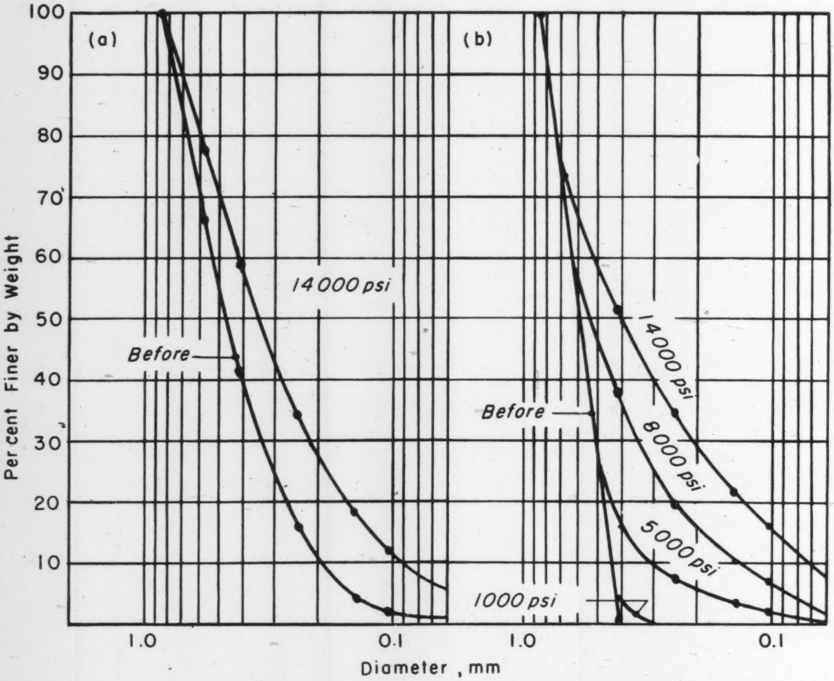
[4] The compression index is equal to $\frac{de}{d(\log p)}$ where e is the void ratio of the clay and p the intergranular pressure. C_c is merely the slope of the compression curve when plotted in the customary manner of void ratio *versus* pressure (log scale).

When the pressure on the sample becomes sufficiently high, however, the load on individual grains becomes large enough to cause shattering. The pressure at which this process becomes evident will be referred to as the "critical pressure." Shattering manifests itself as an increased slope of the compression curves shown in both Figs. 3 and 4. The shattering can easily be heard as a continual "popping" sound as the grains break. This became readily detectable only at pressures in the vicinity of the critical pressure and above. The effects of shattering can be seen from the several grain-size distribution curves shown in Fig. 6(*b*) and from the microphotographs of a sample of No. 20-40 sieve well-rounded material before and after compression to a stress of 6400 psi.

Once the intergranular stress on the sample reaches or exceeds the critical stress, the time lags during compression become important. Figure 5 shows that, for the sample tested, the compression occurring after 10 min is about 20 per cent of the total compression occurring in the 24-hr period. At pressures above the critical pressure the time lag is due to a continuing process: there is a gradual build-up of stress on individual grains resulting in shattering, a redis-



(*a*) Graded Ottawa sand before and after compression.

(*b*) Uniform Ottawa sand before and after compression.

FIG. 6.—Gradation Data for Sands.

tribution of stress occurs followed by a stress build-up and shattering of other grains.

At low pressures the evidence suggests that the importance of the time lag, which is due essentially to a continuous rearrangement of particles, depends on the initial density of the sample. Taylor (17, p. 217) presents a time curve for a typical load increment on a uniform fairly dense sand in which about 95 per cent of the total compression occurred during the first minute. Terzaghi and Peck (22, p. 59) present a time curve for a compression test on a loose sand in which the pressure was increased at a fairly rapid rate and then held constant at 6 kg per sq cm; even after 1 hr, the curve presented indicates that measurable compression was still occurring.

On the basis of the test data presented, it appears that the sands which were initially well rounded have a reasonably unique compression curve above the critical pressure. The pressure at which appreciable breaking of grains occurs is apparently dependent almost exclusively on the initial void ratio. The results of the one test on a graded well-rounded quartz sand suggest that gradation does not have an appreciable effect on the compression index above the critical pressure.

Above the critical pressure, well-rounded quartz has an average compression index of approximately 0.5. It is interesting to note that although the curve for the ground quartz is displaced from those of the rounded grains the compression index of this curve above 700 psi is also approximately 0.5.

It is interesting to compare the slope of the California core curve with the average slope of the quartz sand data obtained in this investigation. The reasonable agreement suggests that these natural sands must have undergone a type of compression similar to that of the test samples above the critical pressure. Unfortunately samples of the cores were not available for microscopic study in order to determine whether fracturing had occurred. In connection with the California cores Terzaghi (21) noted that the median grain size of the sands persistently decreased from about 0.35 mm at the top of the column to about 0.15 mm at the bottom. However, no statement was made regarding the angularity of grains. Still to be explained is the fact that the core samples are considerably more dense than would be predicted on the basis of laboratory compression of pure quartz sand. Possible explanations are: (1) the natural samples have been subjected to the loads for a very great period of time; (2) a process of solution and redeposition might add to the densification of the sands.

CONCLUSIONS AND PRACTICAL SIGNIFICANCE OF INVESTIGATION

From the test data presented the following conclusions can be drawn:

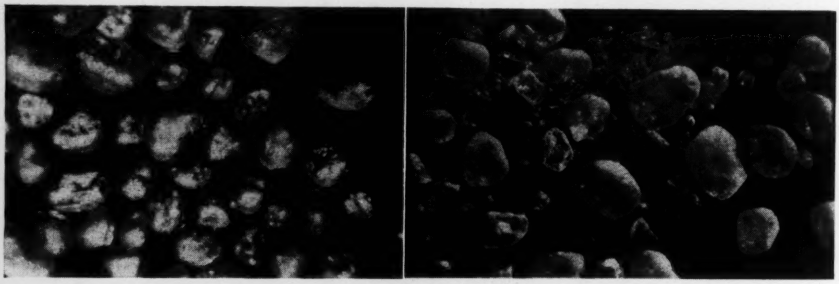
1. At sufficiently high pressures, sand may be more compressible than clay; (test *E* run on ground quartz shows that breakdown pressures as low as several hundred pounds per square inch can be obtained under certain conditions, although it is doubtful that any natural quartz sand as originally deposited contains an appreciable percentage of grains as angular as those used in this particular test);
2. The high compressibility is due primarily to crushing and fracturing of individual sand grains; and
3. A most important factor influencing the pressure at which breakdown occurs for a given initial angularity is the initial void ratio.

The practical significance of these conclusions is that the compression of sands by the weight of overlying sediments is a phenomenon which cannot be neglected by geologists and petroleum engineers. At the high pressures encountered in deep sedimentary deposits, the compression of the sands can be of equal, if not of greater, importance than the compression of clays.

The increase in intergranular pressure brought about by the extraction of oil from deep sand layers may cause large amounts of compression in these layers resulting in not only land subsidence but also augmented recovery of oil.

The fact that most void ratio reduction in sands takes place by a process of fracturing of the grains implies that deeply buried sands with low void ratios almost certainly do not have the same grain-size characteristics that they possessed when first deposited: the median grain-size will tend to be smaller, the angularity more pronounced, and the gradation wider. This should be of considerable importance in the interpretation of the origin and depositional environment of sands, and, as far as is known, this type of post-depositional change has been given little consideration by sedimentologists.

In connection with the interpretation of the history of deep sand deposits it is probable that for those sands which have been subjected to pressures above the breakdown pressure the maximum past pressure can be estimated by means of a compression test in much the same way as is done for clays. The break in the compression curve would indicate the maximum pressure to which the sand had been subjected.



(*a*) 20-40 mesh Ottawa sand before compression. (*b*) 20-40 mesh Ottawa sand after compression.

FIG. 7.—Microphotographs of Sand Grains.

Acknowledgments:

The authors wish to express their appreciation to the Creole Petroleum Corp. for their support of the research described. Special thanks are extended to Karl Terzaghi for permitting us to use the California and Oklahoma core data. Finally, the authors are indebted to T. William Lambe, Head, Soil Engineering Division, Department of Civil Engineering, Massachusetts Institute of Technology, for his continuous encouragement and guidance.

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SUMMARY OF PROCEEDINGS OF THE CONFERENCE ON SOILS FOR ENGINEERING PURPOSES

Mexico City provided a unique as well as highly educational setting for the Conference on Soils sponsored jointly by the Sociedad Mexican de Suelos, A. C. and ASTM Committee D-18 on Soils for Engineering Purposes. The conference consisted of five sessions of papers, listed below, and a meeting of Committee D-18. All meetings were held at the School of Engineering, National University of Mexico in Mexico City, on December 9 to 11, 1957.

The papers, fifteen in number, presented and included in the publication are:

New Developments in Soil Sampling and Rock Coring Equipment—T. W. Van Zelst
Consolidation of Mexico City Volcanic Clay—L. Zeevaert
Laboratory Consolidation Tests for Expansive Clays—R. F. Dawson
Some Factors Affecting the Dynamic Compaction Test—E. Tamez
Compaction Characteristics of Gravelly Soils—W. G. Holtz and C. W. Lowitz
Effect of Tire Pressures and Lift Thicknesses on Compaction of Soil with Rubber-Tired Rollers—W. J. Turnbull and C. R. Foster
A Rapid Method of Construction Control for Embankments of Cohesive Soil—J. W. Hilf
Investigation of a Volcanic Soil for the Construction of an Earth Dam—E. Rios Lazcano
Soil Deformations Under Repeated Stress Applications—H. F. Seed and R. L. McNeill
Unconfined Compression and Vane Shear Tests in Volcanic Lacustrine Clays—R. J. Marsal
Testing Soils with Transient Loads—R. V. Whitman
Correlation of the California Bearing Ratio and the Iowa Bearing Value—D. T. Davidson, R. K. Katti, M. E. Kallman, and John Gurland
Seepage Determination on Hydraulic Structures by Means of Radioactive Isotopes—I. Sainz Ortiz
Sinusoidal Surface Waves in Stratified Soils—J. J. Slade, Jr.
Underpinning and Straightening of an Eleven-Story Building in Mexico City—W. Streu

The papers have been published, together with discussions, in both English and Spanish, as *ASTM Special Technical Publication No. 232* entitled "Conference on Soils for Engineering Purposes."

SUMMARY OF PROCEEDINGS OF THE SYMPOSIUM ON APPLICATION OF SOIL TESTING IN HIGHWAY DESIGN AND CONSTRUCTION

In recent years the expansion of the highway program has placed increased emphasis on the use of soil testing procedures both in design and construction. To present the latest developments in this field Committee D-18 on Soils for Engineering Purposes organized a Symposium at which nine papers were presented dealing with this phase of activity. The paper by R. E. Machol entitled "Laboratory Shear Tests on Varved Clays and Their Application to Design" has not been included in the published volume.

The papers and discussions of the Symposium were presented at the Thirty-seventh and Forty-second Sessions of the Sixty-first Annual Meeting of the Society, held in Boston, Mass., June 26–27, 1958. Mr. C. A. Carpenter, Bureau of Public Roads, and Mr. E. J. Kilcawley, Rensselaer Polytechnic Institute, acted as chairmen of the sessions and Mr. H. B. Seed, University of California was chairman of the Symposium Committee.

The papers presented and included in the publication are:

Introduction—H. B. Seed
Examples of Highway Soil Engineering—E. S. Barber
Experience with Core Drilling Machines, Power Augers, and Electrical Resistivity on the Pennsylvania Turnpike—D. G. Shurig and E. J. Yoder
The Soil Exploration and Mapping Cooperative Project in Illinois—Nicholas Chryssafopoulos
Investigation of Banded Sediments Along St. Lawrence North Shore in Quebec—Robert W. J. Pryer and K. B. Woods
The Value of Soil Test Data in Local and Regional Road Planning—Martin Ekse
Laboratory Methods of Compacting Granular Soils—Earl J. Felt
Ohio's Typical Moisture-Density Curves—J. G. Joslin
Index of Compaction Characteristics—J. L. McRae

The papers with discussion were issued as *ASTM Special Technical Publication No. 239* entitled "Symposium on Application of Soil Testing in Highway Design and Construction."

SUMMARY OF PROCEEDINGS OF THE SYMPOSIUM ON PARTICLE SIZE MEASUREMENT

This Symposium was sponsored by ASTM Committee E-1 on Methods of Testing. Its purpose is to bring up to date the developments in instrumentation and technology that have taken place since the 1941 Symposium on New Methods for Particle Size Determination in the Sub-Sieve Range. Since then, and particularly during the war period, a number of new techniques were developed, some involving new types of apparatus for particle size measurement.

The papers and discussions in this Symposium were presented at the Thirty-first, Thirty-third, and Thirty-ninth Sessions of the Sixty-first Annual Meeting of the Society, held in Boston, Mass., June 26–27, 1958. Mr. L. T. Work acted as Symposium Chairman and Mr. A. E. Reed, the W. S. Tyler Co., Mr. A. E. Jacobsen, National Lead Co., and Mr. Work, Consulting Engineer, presided over the respective sessions.

The papers included in the symposium are:

Sieves, Sieving Methods and Microscopy

Introduction—L. T. Work

The Mechanics of Fine Sieving—K. T. Whitby

A Review of Sieve Standardization (presented by title only)—L. V. Judson

Application of Electroformed Precision Micromesh Sieves to the Determination of Particle Size Distribution of Cracking Catalysts—H. W. Daeschner, E. E. Seibert, and E. D. Peters

A Review of the API Committee on Analytical Research, Subcommittee on Measurement of Physical Properties of Cracking Catalysts—L. L. Mittelman

The Stanford Research Institute Particle Bank—R. D. Cadle and W. C. Thuman

Sedimentation Methods

Recent Developments in the Hydrometer Method as Applied to Soils—E. E. Bauer

Sedimentation Procedures for Determining Particle Size Distribution—W. F. Sullivan and A. E. Jacobsen

A Photoelectric Sedimentation Method for Particle Size Determination in the Sub-Sieve Range—H. R. Harner and J. R. Musgrave

Centrifuge Sedimentation Size Analysis of Membrane Filter Collected Samples of Airborne Dusts—K. T. Whitby, A. B. Algren, and J. C. Annis

A Liquid Sedimentation Method for Particle Size Distributions—L. M. Cartwright and R. Q. Gregg

Methods for Determining Particle Size Distribution by Gravitational and Centrifugal Sedimentation—Soren Berg

Other Particle Size Methods and Correlations

A Discussion of the Recommended Practice of Reporting Particle Size Characteristics of Pigments (ASTM Tentative Method D 1366 - 55 T)—J. H. Calbeck

The Determination of Particle Size by Adsorption Methods—R. J. Fries

A Study of the Blaine Fineness Tester. Calculation of Surface Area from Air Permeability Data—S. S. Ober and K. J. Frederick

Three-Dimensional Electronic Sizing of Sub-Sieve Particles at 6000 per Second—R. H. Berg

Turbidimetric Particle Size Distribution Theory: Application to Refractory Metal and Oxide Powders—A. I. Michaels

These papers with discussion were issued as *ASTM Special Technical Publication No. 234,* entitled "Symposium on Particle Size Measurement."

A LABORATORY METHOD FOR DETERMINING THE SKIDDING RESISTANCE OF BITUMINOUS PAVING MIXTURES*

By J. W. Shupe[1] and W. H. Goetz[2]

One of the major problems currently confronting the highway engineer in many areas of the United States is to contruct highways that will exhibit satisfactory anti-skid characteristics for a reasonable length of time. All pavement surfaces can develop adequate skidding resistance in a dry state. In fact, some of the surface types that are extremely slippery when wet have shown very high skidding resistance in the dry condition (**1, 4**).[3] The problem, therefore, is to design highways that retain good anti-skid characteristics even when water is standing on the pavement surface.

The majority of the highways constructed recently, whether of portland cement or of bituminous materials, have possessed adequate wet skidding resistance when new. Unfortunately, on many of these surfaces this initial anti-skid resistance has been short-lived. As a result, it has become increasingly important to give some consideration to the change in skidding resistance of pavement surfaces due to the polishing effects of traffic.

There is little existing knowledge, other than experience on specific surfaces, with which to predict the long-time behavior of the many different surface types. Those contributions that have been made are the result, for the most part, of field investigations. By testing a large number of highway surfaces and performing a statistical analysis on the results, it is possible to develop some general relationships for the various surface types (**2, 3, 4**).

The skidding resistance of a pavement surface as determined by a highway investigation, however, represents a composite value of a seemingly endless list of variables, including factors pertaining to the aggregate, the binder, traffic and age, and the condition of the surface during testing. It is impossible in a field investigation to evaluate the separate effect of each of these interrelated variables. It was felt that the control and consistency inherent in a laboratory investigation would permit a more accurate evaluation of the basic factors contributing to slipperiness of pavement surfaces. Therefore, laboratory equipment and a testing procedure were developed with this end in mind. Although this paper is devoted entirely to bituminous mixtures, many phases of the testing procedure are equally applicable to portland-cement concrete surfaces, which currently are being investigated in a similar study.

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* Presented at the Sixty-first Annual Meeting of the Society, June 22–27, 1958.

[1] Associate Professor, Kansas State College, Manhattan, Kansas.

[2] Research Engineer, Purdue University, Lafayette, Ind.

[3] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 1305.

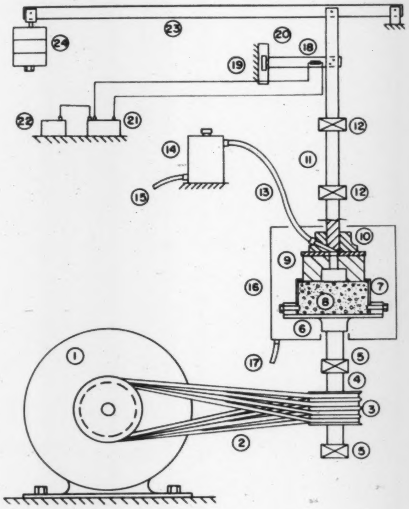

FIG. 1.—Schematic Diagram of Laboratory Skid-Test Apparatus.

DEVELOPMENT OF LABORATORY TESTING EQUIPMENT

There were two separate phases in the development of a satisfactory laboratory method for evaluating the resistance of different types of bituminous paving mixtures to the polishing effects of traffic. The first covered the development of suitable equipment and procedure for measuring the skidding resistance of a test specimen in the laboratory. The second phase, equally important, comprised the selection of a laboratory method, with related instrumentation, for simulating the wear and polishing effect that a pavement surface receives under the action of traffic.

In considering the objectives of the program in the light of previous research, the following requirements seemed essential to the satisfactory determination of the skidding characteristics:

1. The equipment should be adaptable both to specimens molded in the labora-

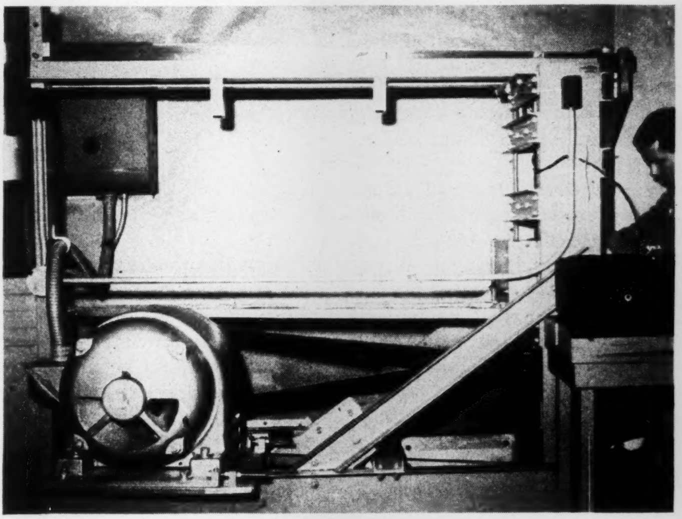

FIG. 2.—Laboratory Skid-Test Apparatus.

tory and to samples removed from the highway surface.

2. Since the degree of wetness is significant, provision should be made to insure that the surface of the specimen is well lubricated during testing.

3. The type of rubber from which the testing shoe is made, the area of contact, and the normal pressure between the testing shoe and the surface of the specimen should be such that the degree of envelopment of the aggregate particles which occurs during braking of a passenger car on a highway surface is simulated.

4. In order to obtain a realistic evaluation, the relative speed between the surface and the testing shoe should be high. On the basis of previous field studies (2), a speed of 30 mph appeared to be a reasonable value.

5. The results should be relatively free from individual bias. To this end, automatic recording would be preferable to some method of dial or scale reading during testing.

6. In order to accomplish a large-scale program of research, the equipment should be rugged and capable of producing a large amount of reproducible data over a prolonged period of testing with a minimum of servicing, adjustment, and repair.

So far as the development of an accelerated wearing and polishing procedure was concerned, the primary criterion for each test specimen was to duplicate, as nearly as possible, the surface characteristics that a similar mix would exhibit after an appreciable amount of highway service under the action of heavy traffic.

The Laboratory Skid-Test Apparatus:

With the objectives listed to serve as a guide, the laboratory skid-test apparatus illustrated in Figs. 1 and 2 was constructed. In the following discussion of the essential features of the skid-test apparatus and the reasoning behind them, reference will be made to Fig. 1, the schematic diagram of the laboratory skid-test apparatus.

The power required to maintain a constant rotational speed of the test specimen during testing is supplied by a 40-hp electric motor, *1*. Anticipated torque requirements were only about one half of this value, but since this heavier unit was available it was incorporated into the design. This did necessitate a sturdier frame than otherwise would have been required, but resulted in an over-all saving when compared to the cost of a smaller motor. It also provided a margin of safety, should subsequent research require a greater torque output.

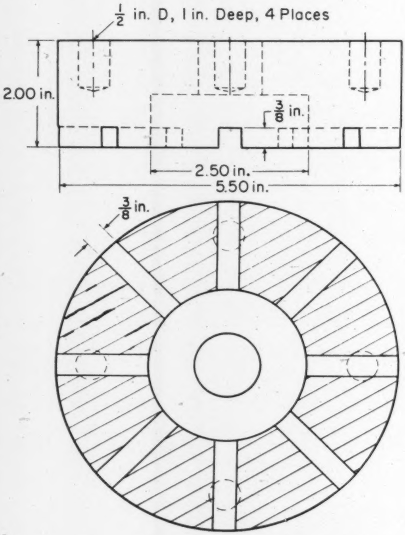

FIG. 3.—Testing Shoe for Skid-Test Apparatus.

The power is transmitted to the lower shaft through four V-belts. The speed of the lower shaft assembly, is approximately 2500 rpm. This rotation results in a relative velocity between the test speciment and testing shoe of slightly over 30 mph at the mean radius of the area of contact.

The lower shaft, *4*, is mounted in two roller thrust-bearings, which are rigidly anchored to the frame of the machine. The bracket, *6*, is fastened to the lower shaft and serves as the base plate for the mold, *7*, and the test specimen, *8*.

The testing shoe, *9*, which is schematically shown in Fig. 3 and illustrated in Fig. 4, is made from rubber disks. The ASTM grade number for this rubber is R630B,[4] and it is representative of passenger car tire tread compounds, having a Shore "A" durometer reading of 65. The testing shoe is anchored to the backup plate by four ½-in. diameter pegs and a rubber-to-metal adhesive of "Loxite-6000."

Referring to Fig. 3, the area in contact with the test specimen consists of eight segments, each having an area slightly over 1¾ sq in., giving a total contact area between testing shoe and test specimen of 14.4 sq in. The vertical force between the two sliding surfaces during testing is maintained at 400 lb, resulting in a unit pressure of about 28 psi. The slots were provided in the testing shoe to permit the free passage of water over the test specimen, to prevent overheating of the specimen or testing shoe, and to simulate, to a limited degree, the tread on the majority of vehicular tires.

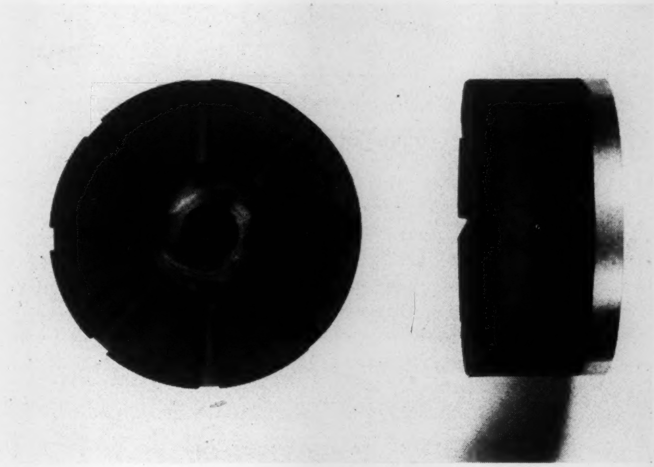

FIG. 4.—Rubber Testing Shoe.

After prolonged testing, the edge of the slot becomes rounded and occasionally, with a particularly abrasive aggregate, the surface of the testing shoe may become scoured. When either of these conditions exist, the testing shoe is removed from the skid-test apparatus and the face redressed. This is done very simply by mounting the shoe and backup plate in a magnetic chuck and grinding off approximately $\frac{1}{32}$ in. of rubber. Before being used again for test measurements the shoe is honed by simulating a test run on a Kentucky rock asphalt specimen.

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[4] Tentative Specification for Elastomer Compounds for Automotive Applications (D 735 – 57 T), 1957 Supplement to Book of ASTM Standards, Part 6, p. 238.

The testing shoe is attached through a backup plate to the upper bracket, *10,* which, in turn, is keyed to the top shaft, *11.* The top shaft is supported in two sleeve bearings which maintain radial alignment of the shoe for all vertical positions. The top shaft assembly does not rotate during testing, and the torque developed in this shaft, due to the sliding friction between the test specimen and testing shoe, is proportional to the skidding resistance of the surface type as represented by the test specimen.

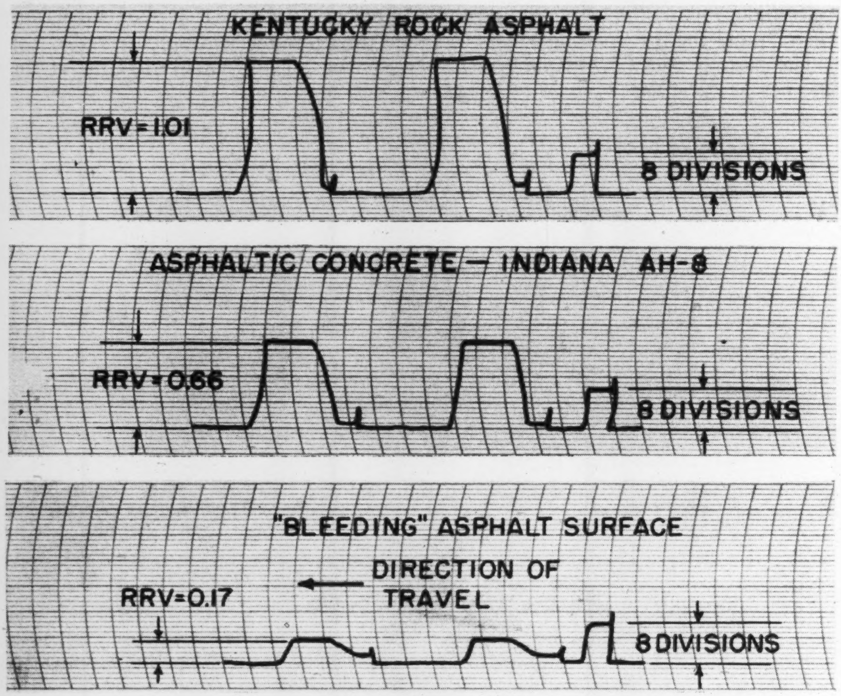

FIG. 5.—Oscillograms from the Laboratory Skid-Test Apparatus.

The torque-reaction moment is developed in a cantilever beam, *18,* on which are mounted two Baldwin SR-4 strain gages. The two gages are located at the vertical neutral axis on opposite sides of the beam and are connected into opposing legs of the Wheatstone bridge circuit, so that any direct force or vertical bending that might be present is not reflected in the torque determination.

In order to ensure that the reaction moment arm maintains a constant length for all vertical positions, a roller-bearing, *19,* is attached to the end of the beam. This bearing makes essentially point contact with the bearing race, *20,* which is a 1-in. diameter steel rod rigidly attached to the frame and parallel to the top shaft.

The strain gages are attached as close to the top shaft as practical in order to take advantage of the largest possible bending moment. The torque developed in the shaft results in a bending moment in the beam, causing a change in resistance in the SR-4 strain gages, which is transmitted to the strain analyzer, *21*, resulting in a deflection of the pen in the automatic recorder, *22*.

Figure 5 illustrates some typical oscillograms for three different surface types. These three test specimens were all cored from highway pavement surfaces, but oscillograms for laboratory specimens are similar in form.

The skidding resistance of each of the surfaces is represented by a numerical value called the relative resistance value (RRV). This skid-test apparatus, like most of the methods for determining the skidding resistance of test surfaces, evaluates the different surface types on a relative basis. No effort was made to convert this reading to a coefficient of friction. Since Kentucky rock asphalt surfaces consistently exhibit the best skidding resistance when wet, as has been determined by previous research (1, 5) attenuation in the analyzer was adjusted so that the deflection of the pen, due to the torque developed by a Kentucky rock asphalt specimen, would be approximately five major divisons. By arbitrarily assigning a value of unity (1.00) to the resistance developed by the Kentucky rock asphalt, the relative resistance values of the other surface types, could be determined to the nearest 0.01.

The three surface types in Fig. 4 show an RRV of 1.01, 0.66, and 0.17, respectively. This represents the maximum range encountered in the testing program, since Kentucky rock asphalt exhibited the best anti-skid characteristics and a "bleeding" asphalt surface, the poorest of all the surface types tested.

Before each of the testing records in Fig. 5 a blip labeled "8 divisions" will be noted. This is the pen deflection caused by the calibration resistance in the analyzer. It was found that by adjusting the a-c gain so that the calibration resistance caused a pen deflection of eight of the small divisions, the deflection resulting from a Kentucky rock asphalt test specimen would be approximately five of the major divisions. Therefore, this 8-division deflection due to the calibration resistance was established as the standard sensitivity, and was adjusted, if necessary, before each test.

Two 3-sec skids were made on each test specimen. The relative resistance value was based on the second trace, as illustrated in Fig. 5. Frequently the RRV for the first skid fell off rather sharply so that there was as much as 0.06 to 0.08 difference in RRV between the beginning and end of the skid. By the second skid, however, the trace was usually sufficiently stable so that an accurate determination of the RRV was possible. On the basis of repeated tests, it was felt an accuracy of ±0.01 could be achieved if care was taken to assure that the 8-division sensitivity was maintained.

The constant load of 400 lb is applied during testing by attaching a load, *24*, of 58.7 lb to the end of the loading bar, *23*. This bar is pivoted at the far end so that a mechanical advantage of slightly over 6 is achieved. This, in addition to the weight of the bar and of the upper-shaft assembly, gives the 400-lb constant normal force required.

The remaining elements of the skid-test apparatus pertain to the water system, which keeps the test surface in a lubricated condition during testing. The water is admitted to the test specimen through a hole located in the center of the testing shoe, *9*, after passing through a passage drilled in the upper-shaft bracket, *10*, and the upper shaft, *11*, as illustrated in Fig. 1.

A flexible water line, *13*, is used so that the system can operate for different vertical positions of the upper shaft assembly. The turbulence chamber, *14*, has no function in the testing operation, but has an important role in the polishing procedure as discussed below. The line pressure maintained at the water inlet, *15*, is such as to result in a rate of flow of water on the test specimen of 7.5 gal per min. All of this water is admitted to the specimen through the center of the testing shoe and flows over the specimen through the slots provided in the shoe.

A shield of $\frac{1}{4}$ in. steel, *16*, is provided to catch the water and channel it to a drain, *17*, and to prevent possible flying particles from causing damage to equipment or personnel.

One testing cycle takes about 15 sec and requires the following operations: (*a*) the motor is turned on, causing the specimen to rotate at 2500 rpm; (*b*) the water valve is opened and the recording equipment started to establish the zero reading on the oscillogram; and (*c*) the weight is lowered manually to allow the full 400-lb force to bear on the test specimen for two 3-sec intervals, during each of which the frictional torque developed in the top shaft is automatically recorded, indicating the skidding resistance of the test specimen.

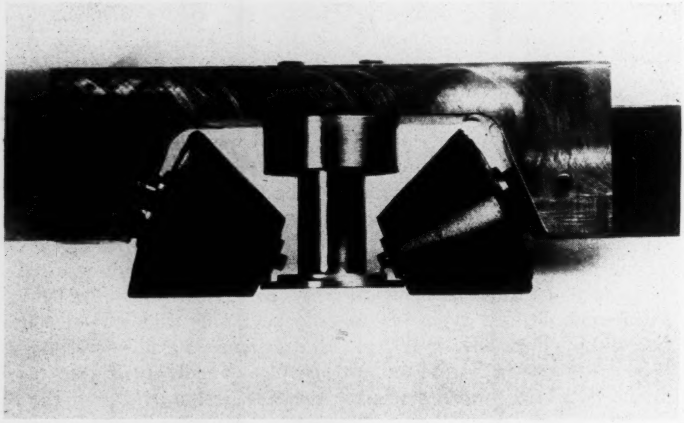

FIG. 6.—Conical Rubber Rollers for Obtaining Adequate Particle Orientation at the Surface of the Test Specimen.

Equipment and Procedure for Simulating Traffic Wear:

The procedure and associated instrumentation for simulating the wear and polishing effect of traffic on pavement surfaces was evolved by investigating different methods of polishing on over 100 test specimens. Various polishing compounds were used including carborundum, alundum, emery, quartz, and limestone varying in particle size from 0.005 to 0.30 mm. Contact pressures ranging from 1 to 28 psi were tried at speeds of 33 and 2500 rpm. Polishing was studied with a rolling rubber cone and with a sliding rubber disk. The length of time for a given polishing cycle was varied from 30 sec to 30 min, with the number of cycles per given specimen varying from 2 to 12.

It was relatively easy to grind the test specimens down using the skid-test apparatus. Referring again to Fig. 1, a charge of abrasive material was first placed in the turbulence chamber, *14*. Then as the valve was opened and water rushed into the chamber from the inlet, *15*, the abrasive was agitated into suspension and flowed down the outlet line, *13*, to the specimen. The inlet pipe was brazed to the circular brass chamber at an angle of 45 deg to the tangent to encourage a swirling action, which would permit adequate transport of the abrasive. Calibration tests showed that with full line pressure at the water inlet, less than 5 g of an initial charge of 500 g of No. 40 to No. 80 sand remained in the turbulence chamber after 60 sec. For limestone mineral filler, less than 1 g was present in the chamber after 60 sec.

The test specimens were ground with a rubber polishing shoe similar in appearance to the testing shoe of Figs. 3 and 4. The outside diameter was $\frac{1}{4}$ in. larger and the inside diameter $\frac{1}{4}$ in. smaller than the corresponding dimensions of the testing shoe in order to ensure that an adequate area would be polished for subsequent testing. Also, the rubber compound from which the polishing disks were made was ASTM grade number R730BJ,[4] slightly harder than that used in the testing shoes.

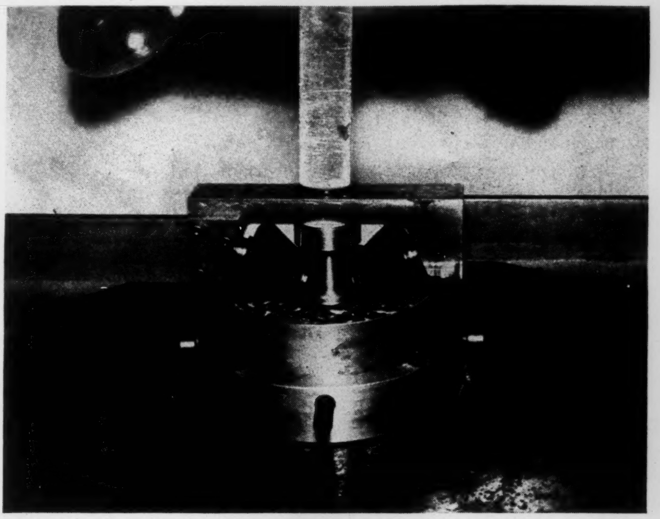

FIG. 7.—Rolling Test Specimen with Conical Rollers in the Minitrack Apparatus.

By starting with a coarse abrasive and a small contact pressure to keep from overheating the polishing shoe, the specimen could be ground down to essentially a level datum plane by decreasing the particle size of the abrasive and increasing the contact pressure as the specimen became progressively smoother. However, a visual comparison of the resulting specimen with a similar mixture cored from the highway showed that there was little similarity between the surface textures of the two. In contrast to the ground specimens, well-worn highway specimens exhibited a texture in which the somewhat rounded and highly polished particles of coarse aggregate projected slightly above the surrounding matrix of asphalt and fine aggregate. This condition could not be duplicated merely by grinding down the specimen.

The conical rollers illustrated in Fig. 6 were intended to supply the rolling action that a surface experiences due to traffic in order to achieve the particle orientation and arrangement that exists on the highway surface. Figure 7 shows a specimen being rolled at 33 rpm in a Minitrack apparatus. This equipment had been developed some years ago for rolling test specimens on a circular track with a small rubber tire. Only slight modification was necessary to adapt the existing equipment to accommodate the 6-in. diameter specimen. Rolling in this manner resulted in an over-all densification of the test specimen, with the coarse aggregate particles becoming somewhat more prominent at the surface than was noted at the completion of the molding procedure.

Rolling was initially attempted with this equipment without the use of the center post. At 140 F some of the mixes shoved excessively and bunched up at the center of the specimen. The post was then incorporated, but located flush with the bottom of the rollers. This eliminated the shoving, but since the post took most of the vertical load, only a slight amount of particle reorientation was accomplished. The final position, as shown in Fig. 6 with the rollers extended $\frac{1}{8}$ in. below the post, represented a compromise in which sufficient confinement was provided without sacrificing an adequate amount of particle orientation.

Another operation which seemed to enhance the effect of the coarse aggregate at the surface was to polish the specimen at 33 rpm in the Minitrack, using the conventional polishing shoe previously described, with limestone mineral filler suspended in water as an abrasive. This operation polished the coarse aggregate particles slightly and also appeared to erode away some of the surrounding matrix of fine aggregate and asphalt so that the resulting surface closely approximated that of a sample removed from the highway surface. It was necessary to give the specimen a final polish in the skid-test apparatus in order to smooth off the rough edges exposed by this operation. This left the test surface in a clean, polished condition.

By rolling the specimen again at 140 F for a short time with the conical rollers, a very light asphalt film could

TABLE I.—GRADATION OF ABRASIVES USED IN POLISHING TEST SPECIMENS.

No. 3/0 QUARTZ

US Sieve	Per Cent Retained
No. 100	0.0
No. 140	6.4
No. 170	9.3
No. 200	18.0
No. 270	18.9
No. 325	18.2
Pan	29.2

No. 5/0 QUARTZ

The only specification for this grade is that 80 per cent will pass a No. 270 US Sieve.

LIMESTONE MINERAL FILLER

State Highway Department of Indiana Specifications

US Sieve	Per Cent Passing
No. 30	100
No. 80	95 to 100
No. 200	65 to 100

The mineral filler used as a polishing agent had over 90 per cent passing the No. 200 sieve.

be made to coat the aggregate. This coating discolored the aggregate somewhat imparting what appeared to be an oily rather than an asphaltic film. It was felt that although this treatment did not exactly duplicate the oil drippings, worn rubber, and debris film often noted on highways during certain seasons of the year (2), the relative resistance values for this final condition would be indicative of the susceptibility of the surface types to this seasonal effect.

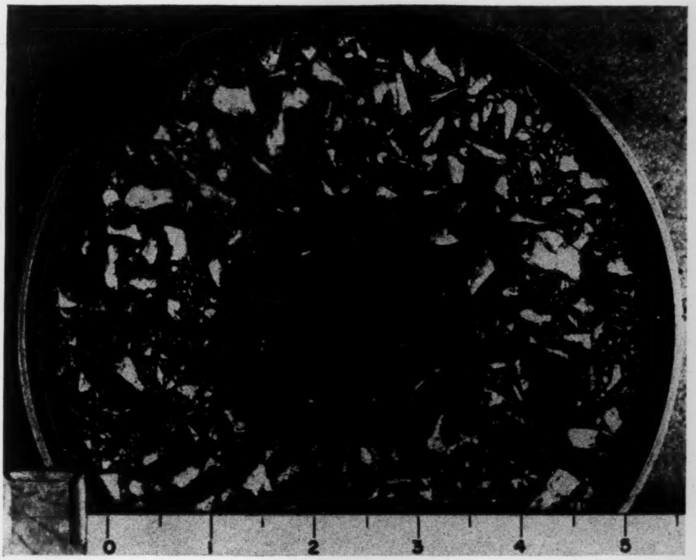

FIG. 8.—Surface Texture at the Completion of the Wearing Procedure for a Limestone Aggregate

The rather complicated wear and polishing procedure as finally evolved consists of the following steps:

1. The specimen is rolled for 2 min at 140 F in the Minitrack at 33 rpm with a total load on the two rollers of 100 lb;

2. Coarse wear is accomplished in the skid-test apparatus with a contact pressure between the shoe and test specimen of 5 psi. Each specimen is subjected to three wear series as follows:

(*a*) 300 g of No. 3/0 quartz[5] for 60 sec,
(*b*) 500 g of No. 5/0 quartz[5] for 60 sec, and
(*c*) 400 g of limestone mineral filler[5] for 60 sec.

3. Particle orientation and texture are developed in the Minitrack at 33 rpm.

(*a*) 2 min rolling at 140 F with a total load of 100 lb on the two rollers, and
(*b*) 10 min of wear, using 10 psi contact pressure with the polishing shoe and 25 g of limestone mineral filler.

4. Final polish is given the specimen in the skid-test apparatus at 2500 rpm.

(*a*) 250 g limestone mineral filler, 15 psi contact pressure, for 30 sec,
(*b*) 250 g limestone mineral filler, 28 psi contact pressure, for 30 sec, and

---

[5] Gradation is listed in Table I.

(*c*) No abrasive, 28 psi contact pressure, for 30 sec.

5. The final operation is to coat the aggregate particles in the specimen surface with a light asphalt film, simulating seasonal road film, by rolling in the Minitrack at 140 F for 1 min with a load of 100 lb on the conical roller.

This procedure was intended to result in a specimen which, both in appearance and in skidding resistance, closely duplicated a similar mix after an appreciable amount of traffic wear; it was not the intention to arrive at the ultimate slippery condition for all surface types. However, the above procedure does cause a specimen made with an aggregate having poor polish-resistant characteristics to approach its most slippery condition. It was felt that by holding the polishing effort constant, a relative measure of the resistance to polishing of the different aggregate types could be obtained that would be more realistic than subjecting each of the test specimens to sufficient polishing to cause it to arrive at its most slippery condition.

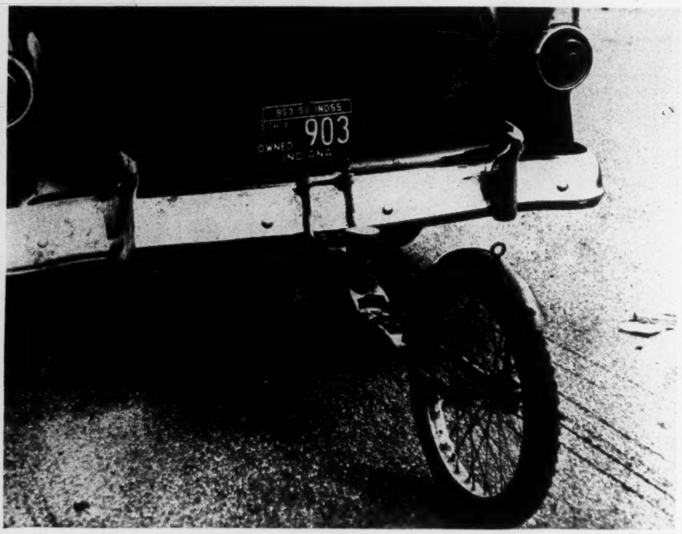

FIG. 9.—The Wagner Stopmeter Attached to the Rear of the Test Vehicle.

It was found that extreme polishing could cause test specimens made from the more-resistant aggregates, such as high-quartz gravel, to have an RRV below 0.4, which is equivalent to a passenger car stopping distance from 30 mph of approximately 150 ft. Since field tests on surfaces of this nature (3) indicate that this material is resistant to polishing, these data did not seem realistic.

During a complete polishing cycle each portion of the test area receives over 80,000 individual passes either from the conical rollers or the separate segments of the testing shoe. Figure 8 indicates the appearance of a typical specimen made with limestone after being subjected to this polishing series.

FIELD CORRELATION STUDY

A field correlation study was made to determine whether the laboratory skid-test apparatus would evaluate the various test specimens in a realistic manner. Passenger car stopping-distance tests were made on selected highway surfaces with the testing equipment of the State Highway Department of Indiana. Cores were obtained from these test sections and the relative resistance values were determined in the laboratory for comparison.

Field Test Equipment and Procedure:

The passenger car stopping-distance test equipment was developed by the Joint Highway Research Project of Purdue University (5), and subsequently transferred to the State Highway Department of Indiana in 1956.

Briefly, the essential components of this equipment consist of a 1955 Ford 2-door sedan equipped with an electrically-operated vacuum braking unit for instantly locking all four wheels, and a Wagner stopmeter (Fig. 9), which is a fifth wheel for indicating both the speed when the brakes are applied and the total distance required to skid to a stop after the brakes are actuated. The vacuum braking system is located in the vehicle luggage compartment.

For this correlation study all skidding tests were performed with the surfaces in a wet condition. A tank truck equipped with spraying apparatus was used for wetting down the surfaces before each test. A crew of five men was supplied by the State for the wetting and traffic control, while the skidding tests themselves were performed by two members of the Purdue staff.

At each of the test sections, the surface was first thoroughly drenched with a slow pass of the watering truck. The test vehicle was then brought to a speed of 30 mph as indicated on the Wagner stopmeter and the brake-actuator button was depressed, causing all four wheels to lock. The distance required for the vehicle to skid to a complete stop, as measured by the Wagner stopmeter, was then recorded. Two more tests of the same section were performed in a like manner with the surface receiving a relatively light water treatment prior to each test.

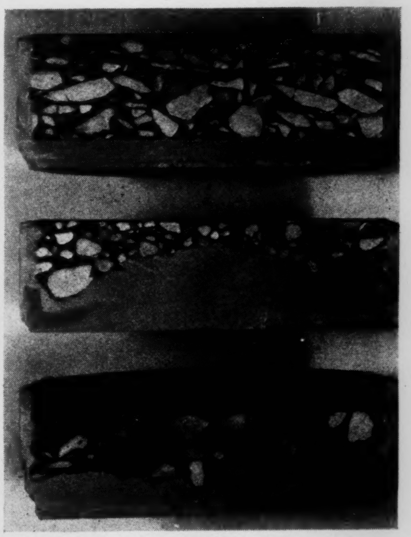

FIG. 10.—Cross Sections of Pavement Cores Mounted in Mortar for Testing.

The top specimen is an asphaltic concrete made with limestone; the middle specimen is from a new asphaltic concrete pavement containing gravel, in which some difficulty was encountered in obtaining a core; and the bottom specimen shows a ½-in. layer of Kentucky rock asphalt on a bituminous-coated gravel base.

On computing coefficients of friction for some of the test surfaces and comparing these values with results of studies in other states, some doubt arose as to the accuracy of the speed measurements. A rough check with a radar speed-determination unit showed that when a speed of 30 mph was indicated on the Wagner stopmeter, the vehicle was actually traveling between 33 and 34 mph. This had no adverse effect on the correlation study, which was merely a comparison of two methods of evaluating the skidding resistance of different test surfaces. However, no direct comparisons should be made between these results and 30 mph stopping distance measurements of other research studies.

Preparation of Test Specimens from Pavement Cores:

Three cores were removed from the pavement from the outside wheel track, at each test section, evenly distributed longitudinally along the length of the skidding area used for the stopping-distance tests.

The cores were drilled with a Model 12, abrasive, speed-drill manufactured by Howe-Simpson Inc. of Columbus, Ohio. The resulting specimen was a core approximately $5\frac{3}{4}$ in. in diameter and, depending upon surface type, anywhere from $\frac{3}{4}$ in. to over 3 in. thick. The bottoms of some of the specimens were extremely irregular. These specimens were mounted in the molds in a paste consisting of three parts of type I portland cement to one part plaster of Paris with sufficient water to provide adequate workability. Figure 10 shows sawed sections of some typical pavement cores.

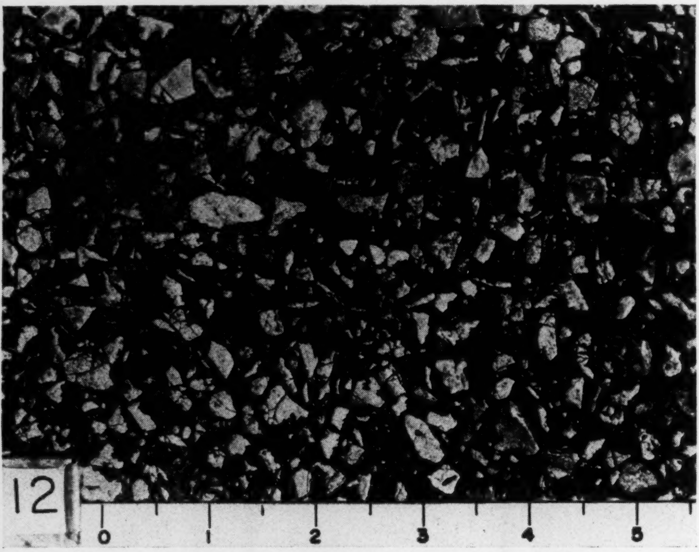

FIG. 11.—Asphaltic Concrete—Limestone.

After the cementing paste had hardened sufficiently, the specimens were tested in the skid-test apparatus in the same manner as for the conventional laboratory test specimens.

Description of Highway Test Sections:

It was not the purpose of this correlation study to evaluate the different

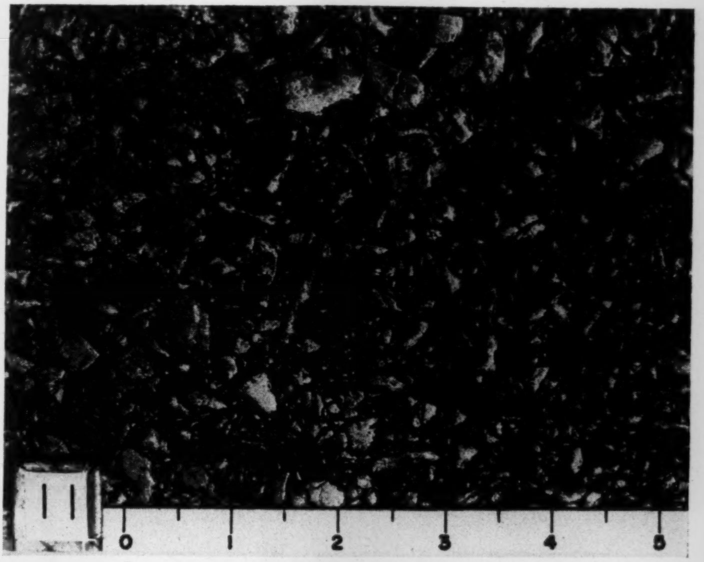

FIG. 12.—Surface Treatment—Limestone.

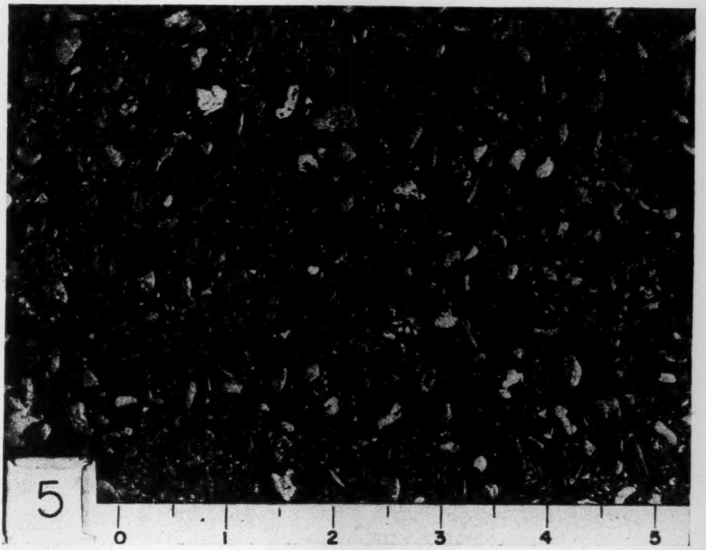

FIG. 13.—Asphaltic Concrete—Gravel.

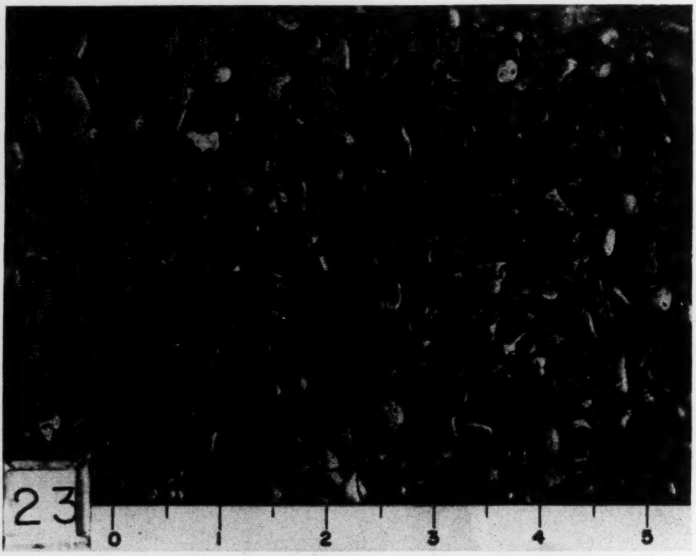

FIG. 14.—Surface Treatment—Gravel.

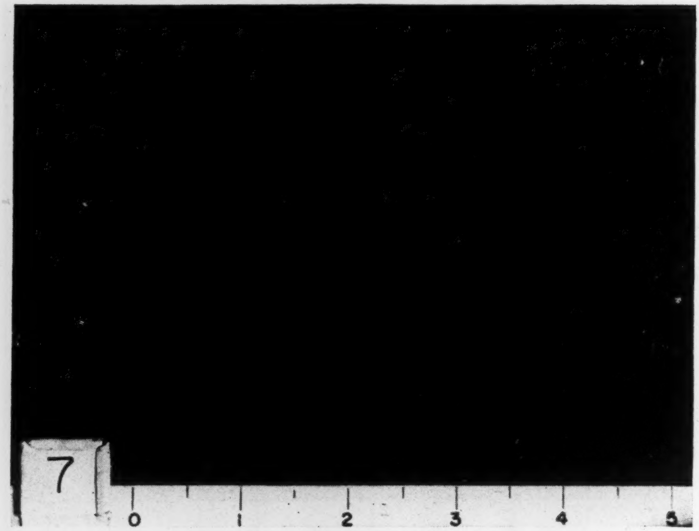

FIG. 15.—Kentucky Rock Asphalt Surface

surface types, as such, and no systematic sampling was made with regard to selecting sections having comparable traffic and age. As a result, no conclusions should be drawn on the basis of these limited data as to the relative skid resistance of the various surface types.

In planning the correlation study, 32 test sections in west-central Indiana were chosen to represent a wide range of bituminous surface types. Three stopping-distance skid tests were made at each location. Examination of the test data led to the selection of 20 of these sections as correlation sites from which three test specimens were to be cored. Because it was impossible at two of these sites to obtain adequate cores, the following correlation study was actually based upon 18 test sections.

Figures 11 to 16 illustrate the six general surface types into which these 18 test sections were classified.

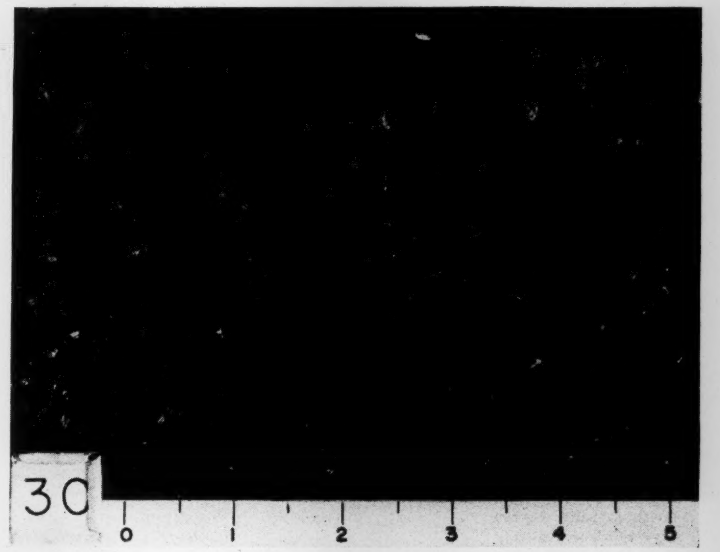

Fig. 16.—"Bleeding" Asphalt Surface.

Figure 11 is representative of those asphaltic concrete surfaces in which the coarse aggregate consisted of limestone. Referring to Table II, test sections 9, 12, and 13 are included in this classification.

Figure 12 is typical of test sections 4, 11, 14, and 26, which are identified rather broadly as limestone surface treatments. All of these surfaces either were given a final surface treatment involving separate applications of asphalt and aggregate, or were of similar texture, so that the resulting area of contact on which the tire skidded consisted almost entirely of coarse aggregate.

Figures 13 and 14 represent, respectively, the same surface types as Figs. 11 and 12, except that gravel rather than limestone was used in these bituminous mixtures. Test sections 3, 5, and 6 are asphaltic concretes made with gravel, and sections 1, 16, 23, and 27 are gravel surface treatments.

Three Kentucky rock asphalt surfaces

were included in this study. This material occurs in Kentucky in a formation in which a Mississippian sandstone is impregnated with asphalt. This type of surface is very uniform and fine-textured, as illustrated in Fig. 15. Test sections 7, 8, and 24 are Kentucky rock asphalt surfaces.

Test section 30 is a "bleeding" asphalt surface located on a secondary county road. Figure 16 is a photograph of this surface.

TABLE II.—SUMMARY OF FIELD AND LABORATORY SKID-TEST RESULTS.

Test Section	Surface Type[a]	Stopping Distance, ft			Relative Resistance Value[b]		
		Average	Maximum	Minimum	Average	Maximum	Minimum
No. 1	G-ST	89.5	90.5	88.5	0.59	0.60	0.58
No. 3	G-AC	99.8	102.0	97.6	0.66	0.67	0.65
No. 4	L-ST	92.5	93.0	92.0	0.57	0.60	0.55
No. 5	G-AC	118.5	120.0	116.5	0.63	0.65	0.62
No. 6	G-AC	83.0	83.5	82.5	0.75	0.75	0.74
No. 7	KRA	69.3	70.0	68.0	0.99	1.01	0.97
No. 8	KRA	62.2	63.2	63.0	1.06	1.09	1.04
No. 9	L-AC	138.0	140.5	135.0	0.45	0.45	0.44
No. 11	L-ST	153.0	153.5	152.5	0.35	0.36	0.33
No. 12	L-AC	85.3	86.1	84.6	0.63	0.64	0.62
No. 13	L-AC	125.3	126.0	124.0	0.53	0.55	0.50
No. 14	L-ST	172.0	176.0	168.0	0.34	0.35	0.34
No. 16	G-ST	103.5	104.0	102.5	0.58	0.60	0.57
No. 23	G-ST	102.0	102.0	102.0	0.55	0.56	0.54
No. 24	KRA	62.0	62.0	62.0	1.01	1.02	0.99
No. 26	L-ST	97.7	98.5	96.5	0.60	0.62	0.58
No. 27	G-ST	93.7	96.5	91.0	0.57	0.60	0.54
No. 30	"Bleeding" Asphalt	Greater than 150			0.23	0.28	0.17

[a] Surface type identification:

L-AC Limestone-asphaltic concrete
L-ST Limestone-surface treatment
G-AC Gravel-asphaltic concrete
G-ST Gravel-surface treatment
KRA Kentucky rock asphalt

Based on a relative resistance value of unity for a Kentucky rock asphalt specimen.

Discussion of Field Correlation Results:

The results for the 18 surfaces on which both stopping-distance measurements and laboratory resistance values were obtained are listed in Table II.

The average figures tabulated in both cases are the arithmetic means of three skids, with the maximum and minimum values also listed to indicate the variability encountered on each of the test surfaces. The stopping-distance measurements are given in feet while the laboratory relative resistance values are dimensionless, but based on a relative resistance value of unity for a Kentucky rock asphalt specimen.

It was impossible to obtain an accurate stopping-distance measurement on test section 30. On each of five attempts the rear end of the test vehicle gradually slid toward the ditch, so that after skidding approximately 150 ft the right rear wheel struck the shoulder and dug in, causing the vehicle to tip appreciably. As a result, the stopping distance on this surface is as listed in Table II as somewhat "greater than 150 ft."

Figure 17 shows the comparison between the field and laboratory data on skidding resistance. Results are plotted for only 15 of the test sections so that an equal number of each of the five general surface types illustrated by Figs. 11 to 15 would be represented. It was impossible to plot the results for the "bleeding" asphalt section.

In order to select a curve that represented the best relationship between the 15 plotted points, a second degree regression curve was computed. However, this curve deviated appreciably from the plotted points at the maximum and minimum values, and it was felt that a visual interpretation would give a better indication of the relationship between the two methods. Therefore, the solid curve of Fig. 17 was drawn in by eye.

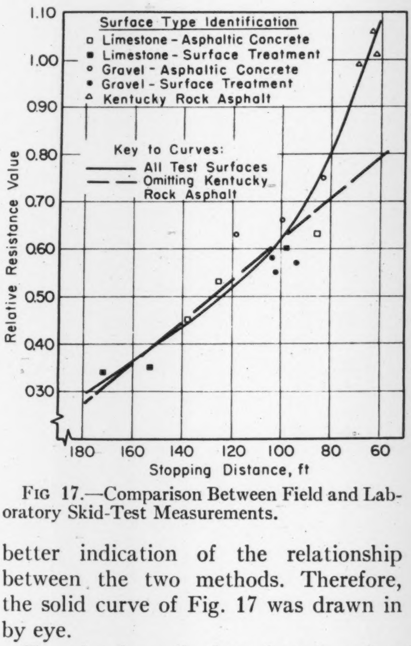

Fig. 17.—Comparison Between Field and Laboratory Skid-Test Measurements.

Examination of the plotted points showed that with the exception of the Kentucky rock asphalt specimens, all of the points conformed fairly closely to a linear plot, thus indicating a linear correlation between the field and laboratory methods of determining skidding resistance. Since there was some justification for omitting the Kentucky rock asphalt surfaces in this analysis, as will be discussed later, a correlation analysis was performed on the 12 remaining test surfaces and resulted in a linear correlation coefficient of 0.86, as computed by the method of least squares (the broken line in Fig. 17).

An examination of the location of the plotted points with respect to the linear correlation line showed that certain surface types consistently fell either entirely above or entirely below the correlation line. This lack of randomness in the location of the plotted points indicated that there was some discrepancy between the field and laboratory methods of evaluation. For surfaces of medium texture there was good agreement in the relative skidding resistance as determined by the two methods. For very open-textured surfaces, however, the laboratory method indicated relatively poorer anti-skid characteristics than did the stopping-distance method. Conversely, for an extremely dense surface the laboratory method showed relatively higher resistance values than were obtained by field measurements.

This relationship is illustrated in Fig. 17 by the location of points corresponding to three different surface types. The very dense Kentucky rock asphalt specimens are all grouped appreciably above the linear correlation line. Similarly the gravel asphaltic concrete surfaces which were quite dense, as shown in Fig. 13, also plotted above the correlation line. However, the open-textured gravel surface treatments, as typified by Fig. 14, resulted in values which fell below the correlation line. The limestone specimens, for the most part, exhibited a medium texture and did not consistently deviate from the linear correlation curve.

Two of the possible reasons why a difference in surface texture caused a discrepancy between the field and laboratory methods of evaluation pertain to the amount of water present on the surface during testing and to the occurrence of a film of oil and dust on the highway test surfaces. The degree of saturation of the test surface was probably the more significant of the two causes. In the laboratory method the rate of water application was constant for all specimens and occurred simultaneously with the test itself, so that the surface was thoroughly drenched during actual testing. In the stopping-distance method, however, there was an appreciable time lapse between the application of the water and the performance of the skidding test. On very dense surfaces the rate at which water drained from the surface was slow, and the test section remained in a drenched condition for a short time. On the open-textured test sections, however, the surface did not remain drenched, particularly when the pavement temperature was well over 100 F, as was the case for the majority of the testing program. It was impossible to maintain a continuous water film on these coarse-textured pavements, and the water quickly evaporated from the tops of the projecting aggregate. As a result, there was not complete consistency in the degree of saturation of the pavement test surfaces. This would tend to result in relatively higher skidding-resistance values for open-textured surface types than for dense surfaces when evaluated by the field stopping-distance method.

The second possible source of difference was the presence of the film of oil drippings, worn rubber, and dust on the highway during testing. Previous research (2, 6) has noted this film as a factor contributing to the so-called seasonal effect, since it is more pronounced during the summer months. It has also been noted that a dense surface is more sensitive to the seasonal effect than an open-textured one. During the coring operation, in which the abrasive and ground-up aggregate swirled around on the surface of the core, and subsequent cleaning of the test specimen, prior to testing, this seasonal film was removed. This resulted in the laboratory test method indicating a relatively higher skidding resistance for a dense surface than for an open-textured surface, as compared to the field method.

A final factor to be considered, which would apply to the Kentucky rock asphalt specimens only, is that previous research (1) has shown that this type of surface possesses nearly uniform high anti-skid characteristics for all speed ranges. The majority of the surface types, however, exhibit a marked decrease in resistance as speed increases (2, 4). The stopping-distance method measures a resistance value which represents an integrated average of the total resistance developed, as the vehicle is skidding from the speed at which the brakes are applied down to zero speed. The laboratory method, on the other hand, evaluates the resistance at a constant speed.

Thus, for a surface which exhibits uniform anti-skid resistance for all speed ranges, the stopping-distance method should give an evaluation of the skidding resistance which would be the same for all speeds, and this value should conform fairly closely to the resistance as determined by the laboratory method. In contrast, a surface type which shows increasing skidding resistance at decreasing speeds should show greater skid resistance by the stopping-distance test (value increasing from 30 mph to 0 mph) than by the laboratory method (values determined at high speed only).

As a result, the stopping-distance method measures a relatively higher skidding resistance than the laboratory method for surfaces which exhibit a decrease in skidding resistance with speed, while the laboratory method indicates a relatively higher resistance for the Kentucky rock asphalt surfaces which possess uniform skidding resistance at all speed ranges. This is illustrated in Fig. 17 by the marked deviation of the points corresponding to the three Kentucky rock asphalt surfaces from the linear correlation line.

In the light of this correlation study, the laboratory skid-test apparatus was considered to be entirely satisfactory for laboratory investigations of factors affecting the skidding resistance of bituminous paving mixtures. Such discrepancies as exist between results by the field and laboratory methods, tend to favor the laboratory method as giving a more realistic evaluation of the skidding resistance of a wet pavement surface for speeds of 30 mph and upward. The laboratory method would probably have shown closer agreement with the truck-trailer method of determining skidding resistance on the highway, since both of these methods evaluate the test surfaces at a constant speed. Unfortunately, equipment of this nature was not available for the correlation study.

PREPARATION OF THE STANDARD LABORATORY SPECIMEN

The primary consideration in the molding of the laboratory test specimens was to arrive at a surface condition which was representative of the texture that a similar mix would exhibit on a highway surface. Although the specimen had to possess sufficient surface durability to prevent disintegration under the severe action of the wearing procedure, stability and density, as such, were not considered important to this study, and no measurements of these properties were made.

Materials:

An 85/100 penetration grade asphalt cement furnished by The Texas Company from the Port Neches refinery was used for all of the test specimens molded in the laboratory. The asphalt content selected for the standard laboratory specimen was 4.5 per cent, based on the total weight of the bituminous mixture. Mixes ranging in asphalt content from 3.5 to 6.5 per cent had been investigated, with the 4.5 per cent figure representing the best compromise. It seemed desirable to keep the mix as lean as possible in order to prevent the asphalt from flushing to the surface during the wearing procedure and obscuring the polishing characteristics of the aggregate, while too low an asphalt content resulted in a mixture which exhibited poor surface stability during the severe wearing procedure.

TABLE III.—INDIANA SPECIFICATION LIMITS FOR HOT ASPHALTIC CONCRETE, TYPE B AND MIXTURE COMPOSITION USED FOR STANDARD LABORATORY SPECIMENS.

Passing Sieve	Retained on Sieve	Type B Limits, per cent		Selected Composition, per cent
		Minimum	Maximum	
½ in.	⅜ in.	2	14	12
⅜ in.	No. 4	20	50	36
No. 4	No. 8	0	22	10
No. 8	No. 16	5	20	11
No. 16	No. 30	5	13	12
No. 30	No. 50	5	12	11
No. 50	No. 100	2	17	3
No. 100	No. 200	1	5	2
No. 200	...	3	5	3
Bitumen		6.5	8.5	4.5

The bituminous-concrete mixture chosen for the standard laboratory specimen conformed to the specifications of the State Highway Department of Indiana for Hot Asphaltic Concrete—type B (medium texture), except for the asphalt content, which was kept low as explained above. Table III lists the specification limits, adjusted to conform to the sieve series used in this investigation, and the selected composition for the standard specimen.

In establishing this gradation as the standard composition, preliminary tests were performed on a wide variety of mixtures ranging in texture from a very dense mix, with the gradation conforming to Fuller's maximum density curve, to an extremely open one which was essentially a one particle size mixture. The asphaltic concrete type B mixture appeared to give the most satisfactory surface texture and degree of coarse aggregate exposure of any of the mixtures tested.

Batching, Mixing, and Molding Procedure:

Most of the mineral aggregates used in this study were received in a crushed condition with the coarse and fine aggregate fractions combined. In order to achieve a high degree of control over the aggregate gradation, the mineral aggregates were first separated into the various sieve size fractions, after which they were recombined in the desired proportions. Three test specimens were made for each mixture, and three different mixtures were molded and tested at a time, giving a total of nine specimens for each series.

In preparing a test specimen, the asphalt and aggregate were heated separately to 300 ± 10 F, and mixed for 2 min in a Hobart electric mixer. The bituminous mixture was than transferred to a mold and vibrated for 1 min with a Cleveland pneumatic vibrator, as illustrated in Fig. 18. On the left side of this figure is a specimen being vibrated showing the collar, bearing plate, bearing block, and vibrator in place, and on the right, a completed specimen.

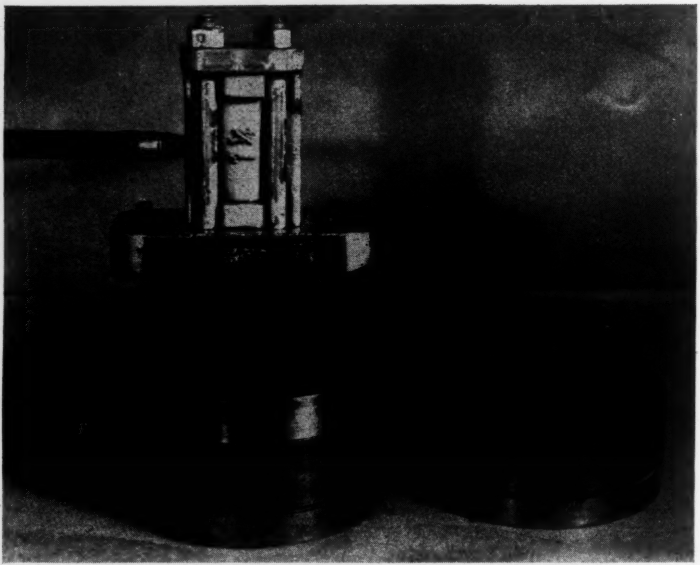

FIG. 18.—Laboratory Vibration Equipment and Test Specimen Mold.

At the completion of the vibration, the specimen was permitted to cool at room temperature for 30 min at which time it was placed in an oven, maintained at 140 F, for an additional 30 min. The specimen was then ready for the first rolling process of the wear and polishing procedure, which is described in a previous section.

RESULTS OBTAINED IN THE LABORATORY INVESTIGATION

Figure 19 illustrates the variation in skidding resistance that a test specimen experiences during an entire wear and polishing procedure. The top curve is for a test specimen made with Kansas sandstone which exhibited the best resistance to polishing of any of the mineral aggregates investigated to date. The middle curve is for a specimen containing Massachusetts rhyolite which, if subjected to a wearing effort appreciably greater than that corresponding to the standard wearing procedure, can be made to polish. The lower curve represents an Indiana oolitic limestone specimen which possesses very little resistance to polishing.

Some of the Indiana limestones tested have shown as high a resistance to polishing as the Massachusetts rhyolite. The limestone, on which the data plotted in Fig. 19 were obtained, is the poorest from the polishing standpoint of any of the Indiana limestones tested. A limestone supplied by another state for this investigation has exhibited even poorer polishing characteristics, however, so the problem of aggregate polishing is not unique with Indiana.

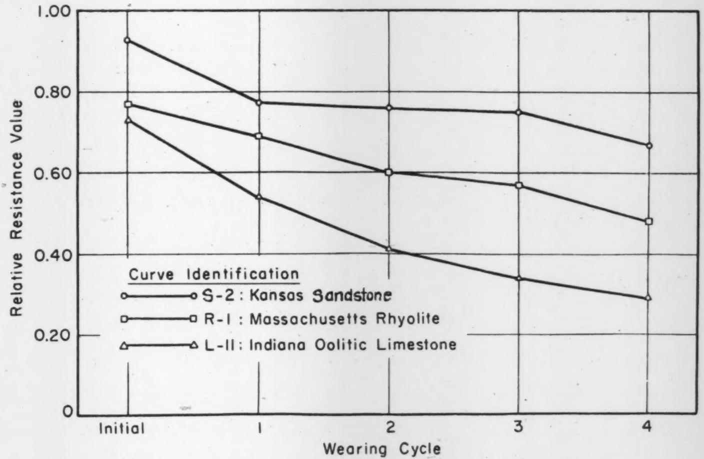

FIG. 19.—Variation in Skid Resistance of Bituminous Mixtures with Wear.

The five points plotted for each of the curves in Fig. 19 correspond to a specific position in the wearing and polishing procedure. The initial relative resistant values were determined after the specimens had been vibrated for 1 min and allowed to cool. The next series was obtained after the specimens had been rolled for 2 min with the conical rollers. The points listed for wearing cycle No. 2 were taken after the specimens were subjected to coarse wear in the skid-test apparatus. The next results correspond to values obtained at the conclusion of the fine-polishing portion of the procedure, and the final points represent the values obtained at the completion of the light rolling operation, which coated the aggregate with a very thin film of asphalt.

In comparing the relative resistance to polishing of the various aggregates, the results obtained at the completion of the fine-polishing procedure are probably the most significant. This corresponds to wearing cycle No. 3 in Fig. 19. At this point in the wearing procedure, the test specimen has a clean, polished surface with a texture quite similar to that of a well-worn highway surface. Subsequent rolling will coat the aggregate and decrease the skidding resistance of the test specimen, but it is debatable as to how realistically this procedure duplicates the traffic film existing on the pavement surface during certain seasons of the year. At the completion of the final polishing, the relative resistance values for specimens made from sandstone, rhyolite, and limestone were 0.75, 0.57, and 0.34, respectively.

CONCLUSION

The laboratory skid-test apparatus has operated satisfactorily for one year, while the wear and polishing procedure, as finally evolved, has been in use for about six months. It appears that this equipment and procedure will prove adequate for a laboratory investigation of factors affecting the skidding resistance of pavement surfaces. Current and future research studies include an evaluation of the effect of the type of aggregate, the particle shape, and the surface texture on the skidding resistance of bituminous surfaces. Investigations will be made for improving the skidding characteristics of bituminous mixtures containing aggregates that polish readily by blending a more resistant material with the polish-susceptible aggregate. Related research will also take place with portland-cement concrete.

Some endeavor will be made to correlate the wearing procedure with actual traffic wear. Although the polishing effort corresponding to the wearing procedure is sufficient to reduce a bituminous surface containing a polish-susceptible aggregate to its most slippery condition, it may prove to be inadequate for duplicating the polishing effect of anticipated traffic on some of our major highways.

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The authors acknowledge the cooperation of the State Highway Department of Indiana in planning and performing the field testing program. W. E. Teske, Research Engineer, Materials and Tests, was particularly helpful in this portion of the study. Morris Stewart of the Joint Highway Research Project at Purdue contributed to all phases of the development of the laboratory equipment and procedure and was in charge of the laboratory testing program. The rubber testing shoes were furnished by the Firestone Tire and Rubber Company.

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FIELD EXPERIENCE AND LABORATORY STUDY OF SLOW SETTING PAVING GRADE ASPHALT IN AIRFIELD PAVEMENT CONSTRUCTION*

By D. D. Fenton[1]

The California State Division of Highways has been conducting studies for a number of years with the aim of developing controls on asphaltic materials that would insure the manufacture of asphaltic products which would have properties superior to those being supplied. During the last part of 1954, a tentative new specification for paving grade asphalts was incorporated into selected paving contracts. After a study of these pavements the new specifications, with minor modifications, were adopted as standard for the California Division of Highways, and the asphalt producers in Northern California modified their manufacturing process to produce paving grade asphalts that would conform to these new specifications. A discussion of the new specifications is contained in a paper presented at the 33rd Annual Conference of the Western Association of State Highway Officials (WASHO) by F. N. Hveem, Materials and Research Engineer, California Division of Highways.[2] The requirements of the new California specifications and the American Association State Highway Officials (AASHO) test methods for paving grade asphalt are shown in Table I.

In the paper presented before the Society the experiences of the Sacramento District of the U. S. Army Corps of Engineers in constructing airfield pavements with asphalts conforming to the new California asphalt specifications are recorded. The materials used, construction methods employed, and the results obtained are discussed and compared with similar airfield pavements placed prior to the change in specifications for paving grade asphalt. For convenience, these pavements are referred to by the contract number under which they were constructed, as follows:

(*a*) *Contract 1286.*—Constructed in 1955, strengthening N.E.-S.W. Runway 4R-22L, Mather Airforce Base;

(*b*) *Contract 1306.*—Constructed in 1955, runway extension, McClellan Airforce Base;

(*c*) *Contract 1354.*—Constructed in 1955, taxiway and lighting, McClellan Airforce Base;

(*d*) *Contract 1167.*—Constructed in 1954, strengthening N.E.-S.W. Runway 4L-22R, Mather Airforce Base; and

(*e*) *Contract 856.*—Constructed in 1953, aircraft storage area, McClellan Airforce Base.

Since there have been some changes in criteria for binder courses during the period 1953 to 1955, the comparison of methods, materials and results will be confined mainly to surface courses.

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* Presented at the Sixty-first Annual Meeting of the Society, June 22–27, 1958.

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[2] F. N. Hveem, "Asphalt Quality Controls," 33rd Annual Conference, Western Association State Highway Officials (1954).

TESTING

All acceptance tests on aggregates and asphalts prior to their use and check tests during construction are made in the South Pacific Division Laboratory of the U. S. Army Corps of Engineers. The results of tests performed on the aggregates, mix design gradations, and optimum asphalt content are summarized in Table II. The results of tests performed on the asphalts used including Abson recovery data are presented in Table III. The Sacramento District maintained a control laboratory at all asphalt mixing plants, during construction, where the gradation of the aggregate and asphalt content of the paving mixture were determined. The penetration of the asphalt was checked and the mixture also checked for moisture content. Tests were also conducted to determine the laboratory unit weight, stability and flow of the material. The per cent total voids and voids filled with asphalt in the laboratory compacted specimens were computed. Specimens of the pavement were obtained each day by cutting cores approximately 4-in. in diameter with a diamond drill. The density of the cores was determined by ASTM Method D 1188 – 56[3] with the exception that the specimens were not coated with paraffin. This was not considered necessary since a dense-graded mix was being used. The density of the cores was compared with those obtained in the laboratory. Data on pavement densities, per cent voids in the total mix, voids filled with asphalt, per cent asphalt, and mix temperatures are shown in Table IV.

TABLE I.—SPECIFICATIONS FOR PAVING GRADE ASPHALTS.

Specification Designation	AASHO Test Method	Grade Asphalt			
		60 to 70	85 to 100	120 to 150	200 to 300
Flash point (Pensky-Martens closed tester), deg Fahr minimum	T73	450	450	425	400
Penetration of original sample at 77 F, per cent	T49	60 to 70	85 to 100	120 to 150	200 to 300
Loss on heating, 5 hr at 325 F, maximum, per cent	California method No. 337	0.85	0.85	1.00	1.50
Penetration after loss on heating, per cent of original minimum	T49	47	45	42	40
Ductility at 77 F after loss on heating, cm, minimum	T51	50	75	75	75
Penetration ratio $\frac{\text{Pen. 39.2 F} - 200\text{ g} - 1\text{ min}}{\text{Pen. 77 F} - 100\text{ g} - 5\text{ sec}} \times 100.$	T49	25 minimum	25 minimum	25 minimum	25 minimum
Saybolt furol viscosity at 275 F, sec	T72	90 to 300	75 to 225	60 to 180	40 to 125
Solubility in carbon tetrachloride minimum, per cent	T45	99	99	99	99
Heptane-xylene equivalent maximum, per cent	California Standard Specifications, section 6, chapter II, article (b-17)	30	30	30	30

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[3] Method of Test for Specific Gravity of Compressed Bituminous Mixtures (D 1188 – 56), 1958 Book of ASTM Standards, Part 4.

The maximum daytime temperatures are presented in Table V.

Construction of Binder and Surface Courses:

The detailed experience encountered in the placing of the binder and surface courses for each of the contracts is omitted from this publication. This information is available at the U. S. Army Corps of Engineers, Sacramento, Calif.

TABLE II.—MIX DESIGN DATA AND TESTS ON AGGREGATES.

Airfield Contract	Mather and McClellan 1286 and 1306			McClellan 1354	Mather 1167	McClellan 856
Year Course	1955 Binder[a]	1955 Binder[b]	1955 Surface	1955 Surface	1954 Surface	1953 Surface
Sieve Size	Mix Design Gradation, per cent passing					
¾ in.	100	100	100	100	100	100
½ in.	84	80	92	94	94	95
⅜ in.			82	84	82	86
No. 4	53	48	66	67	64	64
No. 10	42	35	50	48	52	48
No. 20			35	32	39	33
No. 40	21	18	22	22	28	24
No. 80	14	12	13	14	13	14
No. 200	5.5	4.5	4.5	4.5	4.5	4.5
Mix design bitumen content, per cent	5.6	5.0	6.9	6.5	6.6	6.7

TESTS ON AGGREGATES

Determination	Results			
Abrasion, Los Angeles Rattler, per cent loss	21.5	18	21	19
Fractures, coarse aggregate, 2 faces, per cent by weight	75+	75+	75+	75+
Flats and elongates in coarse aggregate	OK	OK	OK	OK
Swell on test mix, per cent	Nil	Nil	Nil	Nil
Stripping, coarse and fine aggregate	OK	OK	OK	OK
Apparent specific gravities:				
+10 Material	2.776	2.817	2.826	2.774
−10 Material	2.743	2.782	2.726	2.731
Water absorption, per cent	...	0.5	...	...

[a] Original.

[b] Aug. 15, 1955.

DISCUSSION

Aggregates:

The aggregates used in the surface course mixture were American River gravels obtained from deposits in the vicinity of Sacramento, Calif. The coarse aggregate (retained on a No. 10 sieve) was crushed and at least 75 per cent of the particles had two fractured faces. The fine aggregates consisted of crusher "fines" and various blends of sand. The main difference between the fine aggregates used in the various pavement mixtures was that somewhat more crusher "fines" were used in the fine aggregate produced for the work accomplished in 1955. An example of the extreme variation of the gradation of the aggregates, as determined on samples of the mix from which the asphalt had been ex-

TABLE III.—SUMMARY OF DATA ON ASPHALTS.

Airfield	Mather							McClellan				
Contract Year	1286 1955					1167 1954		1306 1955	1354 1955		856 1953	
Producer	A			B		C		A	B	C	A	
Grade	85 to 100		60 to 70	85 to 100	60 to 70	85 to 100		60 to 70	60 to 70	85 to 100	85 to 100	
Course Determination	Binder	Surface		Binder		Surface				Surface		
RESULTS												
Flash point, deg Fahr[a]	450+	450+	450+	450+	450+	450+	450+	450+	450+	450+	460+	450+
Penetration, of original sample, 77 F	97	97	61	90	68	92	91	70	62	91	94	98
Loss on heating, per cent	0.04	0.04	0.0	0.12	0.04	0.1	0.2	0.0	0.0	0.06	0.1	0.03
Penetration after loss, per cent of original	94	94	93	91	91	95	88	91	95	92	99	97
Ductility at 77 F, cm	150+	150+	150+	150+	150+	150+	150+	150+	150+	150+	150+	150+
Solubility in carbon disulfide, per cent	99.8+	99.8	99.9	99.8+	99.8+	99.9	99.8+	99.9	99.9	99.9	99.9	99.9
Organic insoluble, per cent	<0.2	<0.2	<0.1	<0.2	<0.2	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
Softening point, deg Fahr	111	111	116	115	115	112	115	114	119	112	107	111
Spot test	OK	OK	OK	OK	OK	OK	OK	OK	OK	OK	OK	OK
1/8 IN. THIN FILM TEST												
Loss on heating, per cent	0.06	0.06	0.02	0.50	0.2	0.2	0.19	0.05	0.24	0.39		
Penetration, per cent original	68	68	67	56	66	59	56	67	52	55		
Ductility, cm	150+	150+	150+	150+	150+	150+	150+	150+	150+	150+		
Softening point, deg Fahr	115	115	122	128	123	121	120	120	130	130		
ABSON RECOVERY DATA												
Penetration, 1/100 cm	61	63	43	60	44		40			54		
Ductility, cm	150+	150+	150+	150+	150+		150+			150+		
Softening point, deg Fahr	118	117	124	116	123		121.8			126		
Bitumen recovered, per cent	5.2	5.9	4.5	4.7	5.1							

[a] Cleveland open cup test.

TABLE IV.—SUMMARY OF CONTROL DATA.

	Minimum	Maximum	Average	Airfield	Minimum	Maximum	Average	Airfield	Minimum	Maximum	Average	Airfield
	CONTRACT 1286—BINDER COURSE				CONTRACT 1286—SURFACE COURSE				CONTRACT 856—SURFACE COURSE			
Per cent voids, total mix	4.6	6.9	5.7	1955	2.4	5.0	3.7	1955	3.3	5.0	4.3	1953
Per cent voids filled with asphalt	65.5	74.8	68.9	Mather	74.3	86.7	80.3	Mather	75.0	82.0	77.6	McClellan
Asphalt in mix, per cent	5.1 and 4.9	5.6 and 5.2	5.4 and 5.0		6.1	7.3	6.7		6.1	7.2	6.7	
Asphalt mixing temperature, deg Fahr	310	340	320		315	330	320		315	340	330	
Aggregate mixing temperature, deg Fahr	315	350	320		310	350	330		300	335	320	
Completed mix temperature, deg Fahr	295	330	315		310	330	320		305	320	310	
Rolling temperature, deg Fahr	250	285	...		260	300	...		290[a]	...	...	
Relative density in pavement, per cent	96.2	99.2	97.9		95.5	99.9	97.8		96.7	100.8	98.9	
	CONTRACT 1354—SURFACE COURSE				CONTRACT 1167—SURFACE COURSE				CONTRACT 1306—SURFACE COURSE			
Per cent voids, total mix	2.2	3.9	3.2	1955	2.9	5.0	4.3	1954	2.8	4.2	3.6	1955
Per cent voids filled with asphalt	80.1	88.0	83.4	McClellan	75.2	84.2	78.0	Mather	77.6	85.3	81.3	McClellan
Asphalt in mix, per cent	6.5	7.1	6.7		6.2	7.3	6.6		6.2	7.0	6.6	
Asphalt mixing temperature, deg Fahr	300	315	305		295	320	310		310	330	320	
Aggregate temperature, deg Fahr	300	330	320		300	320	315		330	350	340	
Completed mix temperature, deg Fahr	295	320	310		295	315	305		310	330	320	
Rolling temperature, deg Fahr	265	295	...		285	300	...		265	300	...	
Relative density in pavement, per cent	95.1	100.5	97.9		98.0	101.4	99.0		95.0	99.0	97.1	

[a] Data incomplete.

tracted is shown in Fig. 1. The mix design gradations are shown in Table II. All of the mix design gradations fall within a narrow band. The average gradations for the various surface courses, as determined on samples of the mixture from which the asphalt had been extracted are presented in Table VI. These data show no significant difference in gradation. It will be noted that the average percentage passing a No. 200 sieve had a variation of 5.0 to 5.9 per cent. It has been found by experience in the Sacramento District that dense-graded surface course mixtures with this percentage of material passing a No. 200 sieve has good placing properties and may be rolled with a minimum of checking, while mixtures with a high percentage of fines invariably check and present placement problems.

TABLE V.—MAXIMUM DAYTIME AMBIENT TEMPERATURES.[a]

Date	Temperatures, deg Fahr							
	1955				1954		1953	
	June	July	Aug.	Oct.	Sept.	Oct.	Aug.	Sept.
1	...	...	101	88	...	78	...	91
2	...	...	106	...	...	74	...	95
3	...	...	103	...	...	76	...	97
4	...	...	93	78	...	75	...	88
5	...	...	...	78	...	75	...	...
6	...	...	...	83	...	74	...	...
7	...	...	...	85	...	78	...	...
8	...	...	...	...	...	78	...	...
9	...	...	...	...	...	81	...	...
10	...	...	...	64	...	80	...	...
11	...	94	...	...	...	84	...	...
12	...	96	...	...	...	85	...	...
13	...	...	...	...	86	79	96	...
14	...	...	...	...	78	88	85	...
15	...	...	92	...	79	91	86	...
16	...	...	87	...	77	75	91	...
17	...	...	92	...	77	77	94	...
18	...	...	92	...	83	76	95	...
19	...	93	97	69	85	75	91	...
20	...	91	99	75	90	78	87	...
21	...	92	...	75	82	85	87	...
22	...	93	...	...	76	...	96	...
23	...	...	...	...	87	...	90	...
24	88	...	79	...	94	...	87	...
25	84	86	82	85	96	...	81	...
26	...	85	88	...	97	...	82	...
27	...	89	93	...	91	...	79	...
28	...	93	...	...	82	...	75	...
29	...	99	94	...	82	...	75	...
30	...	...	98	...	72	...	82	...
31	...	...	102	...	...	...	88	...

[a] As recorded by the U. S. Weather Bureau at Sacramento.

Mixing Plants and Mix Temperatures:

Continuous-flow and weigh-batch plants were used to produce the surface course mixtures. Both types are considered satisfactory.

The extreme range of temperatures for the asphalt, aggregate and completed mix are shown in Table IV and the average values are presented in Table VI. These data show that the average aggregate temperatures were slightly higher for the work accomplished in 1955, while the asphalt temperatures and temperature of the completed mixes were all comparable. The average aggregate temperature and the average temperature of the completed mix for contracts 1354 and 856 are identical, while the asphalt temperatures for these contracts were 305 and 330 F respectively. The temperatures of the asphalt, aggregate, and the completed mix for all of the contracts complied with the specifications requirements. It has been found by experience in the Sacramento District that the temperature of the completed mix should be approximately 300 F to insure proper coating of the aggregate with asphalt. No difficulties were encountered in 1953 with the placement of mixes with temperatures of 320 F. The temperatures of the asphalt, aggregate and completed mixes recorded in this report should not cause significant differences in the placement of the mixtures provided that specification requirements are complied with.

Asphalt:

The paving grade asphalt used in airfield pavements are specified to meet the requirements of Federal Specifications SS-A-706b which include the following: (1) Flash point (ASTM Method D 92 – 57),[4] not less than 347 F, (2) Softening point (ASTM Method D 36 – 26),[5] from 113 to 149 F, (3) Penetration at 77 F (ASTM Method D 5 – 52),[6] 85 to 100, (4) Loss on heating, 5 hr, 325 F, (ASTM Method D 6 – 39 T)[7] not more than 1 per cent, (5) Penetration after loss on heating, per cent of original, not less than 60, and (6) Solubility in carbon disulfide, not less than 99.5 per cent.

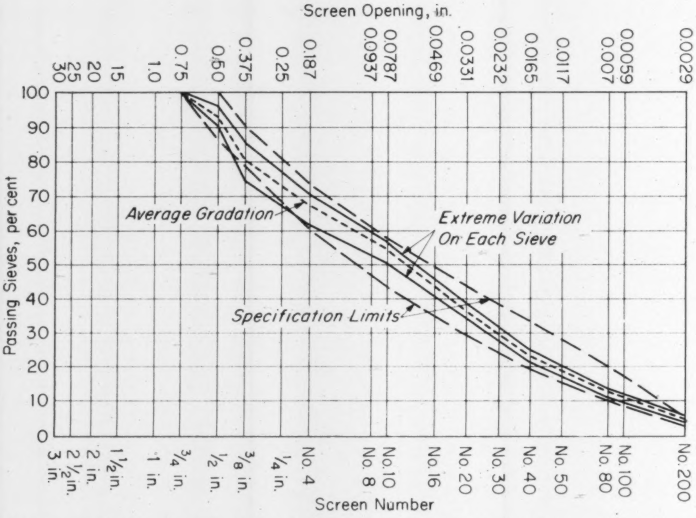

FIG. 1.—Aggregate Grading Chart. Surface course, gradation data from extractions. Contract No. 1286 (1955).

In addition, the asphalt cement is required to show a negative spot when subjected to the test specified in the AASHO test (T102) using standard naphtha. In 1954 the additional requirement was added that asphalt be tested in accordance with the Thin-Film Oven Test procedure set forth in the U. S. Bureau of Public Roads publication *Public Roads*[8] and when so tested meet the following requirements: (1) Loss of weight shall not exceed 1 per cent, (2) Penetration shall not be less than 50 per cent of that of the original asphalt, and (3) Ductility shall not be less than 100 cm.

All of the asphalts used in paving mixtures discussed in this report met all of the physical and chemical requirements of the Federal Specifications and project specifications. However, the asphalts used in 1955 did not produce paving mixtures with the same characteristics as the mixtures used previous to 1955. The results of Abson data on asphalt recovered from the pavement showed that the characteristics of the asphalt were different from those of the material used previous to 1955. The penetration of 85 to 100 grade asphalt recovered from the pavements placed prior to 1955 was of the order of 40 while the penetration of 85 to 100 grade asphalt recovered from pavement placed in 1955 was of the order of 60. The South Pacific Division Laboratory was requested to conduct a study to determine, if possible, what caused this difference. In the course of this study a determination of the percentage of oily constituents was made on samples of various asphalts as they were received and after they had been subjected to the Thin Film Test. The results of these determinations, with the Division Laboratory comments are presented in Table VII.

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[4] Method of Test for Flash and Fire Points by Cleveland Open Cup (D 92 – 57), 1958 Book of ASTM Standards, Part 4.

[5] Method of Test for Softening Point of Bituminous Materials (Ring- and Ball-Method) (D 36 – 26), 1958 Book of ASTM Standards, Part 4.

[6] Method of Test for Penetration of Bituminous Materials (D 5 – 52), 1958 Book of ASTM Standards, Part 4.

[7] Tentative Method of Test for Loss on Heating of Oil and Asphaltic Compounds (D 6 – 39 T), 1958 Book of ASTM Standards, Part 4.

[8] Vol. 27, No. 9, Aug., 1953, p. 202.

TABLE VI.—SUMMARY OF CONTROL DATA ON SURFACE COURSES.

Air Base Year Contract Number	Mather 1955 1286	McClellan 1955 1306	McClellan 1955 1354	Mather 1954 1167	McClellan 1953 856
AVERAGE GRADATION					
Per cent passing:					
¾ in. sieve	100	100	100	100	100
½ in. sieve	93	96	90	96	96
⅜ in. sieve	80	78	80	84	87
No. 4 sieve	67	66	65	65	64
No. 10 sieve	54	52	46	52	50
No. 20 sieve	36	35	32	42	35
No. 40 sieve	23	23	23	26	24
No. 80 sieve	12.1	12.2	13.5	10.6	13.1
No. 200 sieve	5.2	5.0	5.9	5.3	5.2
Average per cent asphalt	6.7	6.6	6.7	6.6	6.7
AVERAGE MIXING TEMPERATURES, DEG FAHR					
Aggregate	330	340	320	315	320
Asphalt	320	320	305	310	330
Completed	320	320	310	305	310
RELATIVE DENSITY, PER CENT					
Minimum	95.5	95.0	95.1	98.0	96.7
Maximum	99.9	99.0	100.5	101.4	100.8
Average	97.8	97.1	97.9	99.0	98.9
DISTRIBUTION RELATIVE DENSITY TESTS					
Range of Relative Densities	Per Cent of Tests in Indicated Range				
Less than 95 per cent	0	0	0	0	0
95.0 to 95.9	5	21	15	0	0
96.0 to 96.9	5	21	3	0	1
97.0 to 97.9	54	29	34	0	5
98.0 to 98.9	28	21	27	55	56
99+	8	8	21	45	38
Per cent of densities 98 per cent +	36	29	48	100	94

In general the Northern California refineries produce various grades of paving asphalts by fluxing back a hard asphalt such as 40 to 50 penetration with lighter grade asphalt such as 200 to 300 penetration or by the addition of selected lube stocks. It seems evident that the asphalts supplied in 1955 for the contracts under discussion were manufactured by this process. Asphalt of 85 to 100 penetration from producer *A* was used in 1954 to produce the paving mixtures for contract 1167 and the Abson recovery data showed that the asphalt recovered from the pavement had a penetration of 40, while the Abson recovery data showed that 85 to 100 penetration asphalt from producer *A* used in 1955 had a penetration of 61 in the pavement. It is doubtful that a crude material distilled to 85 to 100 penetration would have as wide a variation in penetration values in the pavement as indicated above.

TABLE VII.—ESTIMATION OF OILY CONSTITUENTS IN PAVING GRADE ASPHALT.

Manufacturer	Date of Sample	Project	Grade of Asphalt	Per Cent Oily Constituents in Sample After Lewis Thin-Film Test
C.	Dec., 1954	Davis-Monthan	85 to 100	56
D.	April, 1955	Edwards	85 to 100	49
B.	Oct., 1955	McClellan	60 to 70	60
C.	Jan., 1956	Ft. Huachuca	85 to 100	45
A.	July, 1955	Mather	60 to 70	62
A.	July, 1955	Mather	85 to 100	60

The oily constituents were determined after a method given by Abraham on p. 1230 of the work "Asphalt and Allied Substance" (Fifth Edition). The results give an indication that the product supplied to Mather Air Force Base in July, 1955 was high in oily constituents when compared with asphalt manufactured in Southern California. These oily constituents, in the *A* product, were nonvolatile at 325 F, and were of a nature which imparted slow-setting characteristics to the asphalt which made it very difficult to roll the pavement to specified density under normal rolling operations.

As stated before, all of the asphalts used to produce paving mixtures for the contracts discussed in this report met all of the chemical and physical requirements of the Federal Specification SS-A-706b. However, it appears that a purchaser of asphalt specifying that he be supplied with asphalt conforming to the above Federal Specification has no assurance that he will be supplied with asphalt having the characteristics that he anticipates based on past experience. In addition to the physical and chemical properties and requirements Federal Specification SS-A-706b contains the following:

> "C.—*Materials and Workmanship*
>
> C-1.—The materials under this specification shall be asphalts prepared by the distillation of asphaltic petroleum or by the fluxing of hard native Trinidad asphalt with a suitable petroleum flux."

It is not believed that it was the intent of the writers of this specification that various grades of asphalts be produced by fluxing hard and soft asphalts except in the case of native Trinidad asphalt. In a report to the Chief of Engineers[9] E. F. Kelly, Chief, Physical Research Branch, Bureau of Public Roads, made the following statement:

> "Therefore, it is doubtful, regardless of the quality of the asphalts produced by the Propane Deasphalting Process, *or by the process of blending two penetration grades, one hard and the other soft prepared by normal refining practice, or by other means that do not provide for direct distillation to penetration grade; that such asphalts meet the general requirements of the standard specifications or the intent of the writers of these specifications.*"

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[9] *Public Roads*, No. 90010.

Placing:

The equipment used to place all of the surface course mixtures was essentially the same. The operation of the rollers was very much the same except for those mixtures placed in 1955 when various rolling procedures were tried to attempt to overcome the excessive movement of the pavement. The chief difference in rolling operations between the work accomplished in 1955 and the work prior to 1955 was the extensive use of the 10-wheel trucks in 1955. In the Sacramento District the initial breakdown of the paving mixtures, in general, is made when the mixture has a temperature of 285 to 300 F and good results have been obtained. In 1955 the initial breakdown was tried at temperatures varying from 250 to 300 F. Little difference was noted when the breakdown was made at any temperature in this range. It should be noted that the use of 10-wheel trucks to increase the density of the pavement had to be delayed to 4 days after the pavement was placed to be effective and that the density could be increased by truck rolling as much as 13 days after placing. Asphaltic concrete placed prior to 1955 could be rolled immediately after it was placed and would not displace when rolled with trucks. The mixtures placed prior to 1955 contained 85 to 100 penetration grade asphalt and the type and grading of the aggregate was essentially the same. Since the asphaltic cement used in 1955 was different than that previously used and the resultant asphaltic concrete mixture required 4 days before it could be rolled with trucks it has been designated as slow-setting asphalt. The results of density determinations, expressed as a percentage of the laboratory density, for such contracts are summarized in Table VI. These data indicate that the poorest results were obtained on contract 1306 where the surface course was placed immediately after the placement of the binder course. In addition the experience gained in 1955 indicates that airfield pavements constructed with asphalt that has slow-setting properties should not be used by aircraft with high tire pressures until the pavement has been in place for at least one month and perhaps longer. The maximum ambient temperatures during the time the pavements were being placed in 1955 are considered to be normal. It was found while placing the surface course for contract 1354 that normal rolling procedures resulted in satisfactory pavement densities when the ambient temperature was 78 F or less.

The relative density data summarized in Table VI shows that excellent densities were obtained for those mixtures placed prior to 1955. Even though considerably more compactive effort was used in 1955, the results obtained are considered as poor.

CONCLUSIONS

It is concluded that:

(*a*) The quality and gradation of the aggregates used in the paving mixtures were satisfactory for the production of dense-graded asphaltic paving mixtures for airfield pavements,

(*b*) The temperature of the aggregate, asphalt and completed mix for all contracts complied with the specification requirements and were satisfactory,

(*c*) The equipment used to place and compact the paving mixtures was adequate to produce pavements with a high relative density,

(*d*) The asphaltic cement used in all mixtures complied with the physical and chemical requirements of Federal Specification SS-A-706b and the project specifications,

(*e*) The asphaltic cement used in 1955 had slow-setting properties,

(*f*) The asphaltic cement used in 1955 was not satisfactory for use in the manufacture of airfield pavements with the present density requirement, and

(*g*) Asphalts produced by blending two grades of asphalt do not comply with the intent of the Federal Specification.

EFFECT OF ULTRAVIOLET LIGHT ON VISCOSITY OF THIN FILMS OF ASPHALT CEMENTS†, *

By Richard F. Sparlin[1]

Synopsis

The hardening of thin exposed films of asphalt in service is in part due to the effect of ultraviolet energy. Asphalt durability studies by others have indicated that ultraviolet energy acts as a catalyst, accelerating oxidation and consequent hardening of the asphalt. The purpose of this investigation was to determine whether or not ultraviolet energy is capable of hardening asphalt when oxygen is not present.

Thin films of two asphalt cements were exposed to ultraviolet energy while held between glass slides. After exposure the asphalt was sheared in a microviscometer and the viscosity computed. The results were compared by statistical methods to viscosities of control samples.

Though the number of tests conducted was quite limited, covering only a few samples of the two paving asphalts, the results indicate that ultraviolet energy is capable of producing measurable increases in the viscosity of asphalt films sealed from the atmosphere.

Asphalt is a dark, highly viscous material consisting of hydrocarbons and their derivatives. It is a residue of certain kinds of petroleum after the evaporation of more volatile constituents, a process which may take place in nature or be induced by artificial means. Asphalt has many uses in present day construction: for example as a binder in highway pavements, a protective coating on drainage pipes, or as a binder to hold mineral aggregate to the felt of roofing material.

In these applications and many others, the asphalt is distributed in films, some of which are so thin as to be transparent. Regardless of the film thickness, asphalt makes an excellent binder because it is adhesive, cohesive, self-healing, resistant to abrasion, and waterproof. With the passage of time, however, the asphalt cement will suffer a gradual loss of these desirable properties.

This lessening of effectiveness is caused by hardening which is the result of continued exposure to heat, light, air, and moisture, which are ever present in the environment. A review of the literature reveals that many investigations have been concerned with the effect of ultraviolet light in accelerating the hardening of asphalts.

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† This paper presents the results of an investigation concerning the hardening of asphalt cement, and reports the results of limited laboratory experiments conducted during 1957 under the auspices of the Graduate School of Cornell University. The work was directed by Taylor D. Lewis of the School of Civil Engineering and was made possible by a grant from the Esso Standard Oil Co. of New Jersey.

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Ultraviolet light is present to a limited extent in natural sunlight, and it is known to enter actively into many photochemical reactions, but the exact manner in which it effects asphalt is not known.

This paper presents the results of an investigation undertaken to determine the effect of ultraviolet light upon the hardening of films of asphalt sealed from the atmosphere and held at room temperatures so that the effect of oxygen, heat, and moisture may be neglected, and any resultant change in properties could be attributed to the absorption of radiation. This was done by irradiating thin films held between microscope slides for periods up to ten days, and then shearing the asphalt in a microviscometer to determine any change in the rheological properties. For the purposes of this report, the hardness of an asphalt was characterized by its viscosity.

HARDENING

The hardening of an asphalt binder is usually discussed in terms of the separate reactions that occur in service and result in a progressive loss of desirable properties. The reactions are:

1. Oxidation,
2. Volatilization,
3. Thixotropy,
4. Syneresis, and
5. Polymerization.

Oxidation is the combination of the hydrocarbon compounds of asphalt with oxygen and results in a direct union, with the elimination of a portion of the hydrogen and carbon. Abraham (1)[2] has characterized these reactions as follows:

$$C_xH_y + O \rightarrow C_xH_yO \quad \ldots\ldots\ldots\ldots (1)$$

$$C_xH_y + O \rightarrow C_xH_{y-2} + H_2O \quad \ldots\ldots (2)$$

$$C_xH_y + 2O \rightarrow C_{x-1}H_y + CO_2 \quad \ldots\ldots (3)$$

These reactions (Eqs 1, 2, and 3, in that order), require progressively greater amounts of activating energy (heat and light) to promote them, and it is doubtful if the reaction of Eq 3 occurs even at elevated mixing temperatures. An oxidation reaction of type 1 occurs mainly on the exposed surface of the asphalt. It forms a protective skin on the outer surface, and if this protective cover is not washed off, the rate of oxygen absorption slows and hardening is retarded (2).

Volatilization is the gradual loss of lower molecular weight constituents of the asphalt by evaporation and results in a harder material. The rate of evaporation depends upon the ambient temperature, the surface area exposed, and air movement near the surface.

Thixotropy is a flow property of a colloidal system characterized by the ability of the system to undergo a reversible isothermal change in structure from a solid to a liquid on the application of sufficient shearing force. After removal of this force, the liquified system regains its rigid characteristics (3). Many investigators have reported stored asphalts to be thixotropic in character, developing a structure with the passage of time. This internal structural change develops rapidly at first and then proceeds at a steadily decreasing rate (4). There is some indication that air-blown asphalts time-harden faster and to a greater extent than steam- or vacuum-refined asphalts under identical storage conditions (5).

Syneresis is defined as: "An exudation reaction occurring in asphalt, in which certain heavy oils, normally acting as plasticizing oils (keeping the asphalt soft and alive), become incompatable with the basic asphalt structure and are forced to the surface of the asphalt where they are lost (6)."

Polymerization is a molecular condensation process in which two similar molecules combine to form a new heavier molecule having properties different from the two parent molecules. In asphalt, it results in a harder, less flexible material.

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[2] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 1324.

WEATHERING

In general, it is agreed by most investigators that the hardening of asphalt binders in service is due in large part to oxidation and volatilization, and to a lesser degree to thixotropy, syneresis, and polymerization. It is also generally accepted that oxidation is accelerated by radiant energy, either in the form of heat or light. In view of this, it is reasonable to expect that mechanical devices which simulate the effects of natural weathering (weatherometers) contain radiant energy sources. Most of them do, having lamps which furnish energy in either the ultraviolet or infrared range, or both.

Infrared energy is found in the longer wavelength portion of sunlight and constitutes approximately 50 per cent of the total. Ultraviolet energy is found on the other end of the sun's spectrum. Its wavelengths are much shorter and it constitutes only about 5 per cent of the total, the remaining 45 per cent being visible light.

The following brief review of other investigations serves to illustrate the lack of agreement between investigators regarding the effects of ultraviolet and infrared radiation.

Zapata (7–9) has studied the suitability of infrared and ultraviolet radiation for use in accelerated weathering tests. He reports infrared radiation to be preferable to ultraviolet radiation since the intensity of ultraviolet radiation varies throughout the year, and it is almost completely stopped by an asphalt film 1 mm thick (9).

Stanton and Hveem (10) propose the concept that infrared radiation, since it can penetrate into a mix, is probably more of a contributor to aging than ultraviolet radiation. They used a series of infrared drying lamps, enclosed in a weatherometer, and found that considerable deterioration of asphalt could be produced by the action of infrared lamps alone.

Ultraviolet radiation has also been the subject of much discussion and review. Thurston and Knowles (11), in an exploration of susceptibility of the components of asphalt to oxidation, concluded that oxidation at 140 to 185 F is primarily due to the photocatalytic effect of ultraviolet light. This fact was demonstrated by parallel tests on shielded and exposed asphalt samples in which the former absorbed 6 ml while the latter absorbed 26 ml of oxygen.

TABLE I.—ASPHALTS AGED ON GLASS PLATES.

	Type of Asphalt	
	California 85–100 penetration	Venezuela 85–100 penetration
Aging index[a], n/n_o		
Aged 94 hr in 140 F oven, dark	3.60	2.75
Aged 330 hr in 140 F oven, dark	8.41	4.64
Aged 15 min in 140 F W,[b] light	1.78	3.32
Aged 30 min in 140 F W,[b] light	3.20	5.50
Aged 60 min in 140 F W,[b] light	8.11	10.01

[a] The aging index is the ratio of the aged viscosity, n, to the original, n_o.

[b] Twin arc weatherometer.

Weetman (12), in a study of factors affecting accelerated weathering tests, concluded that ultraviolet light acts as a catalyst and that comparable weathering values will be obtained as long as some ultraviolet energy is left in the spectrum.

In a recent study of the factors influencing weathering of paving asphalts, Vallerga (13) found that weathering thin films in the presence of ultraviolet radiation caused substantial reduction in the penetration of the asphalt samples. Similar tests using infrared radiation, however, failed to produce as much drop in penetration.

Table I shows data from a paper by Griffin, Miles, and Penther (14). The table gives the rapid hardening effect of ultraviolet radiation on films 5μ thick for two paving asphalts. These asphalts were aged on glass slides with the film surfaces exposed to both the air and the radiation. Viscosity data were obtained by the use of the sliding plate microviscometer recently introduced by the Shell Development Co.

In an investigation to determine the effect of sunlight on bitumen used for coating tin roofs in Germany, Von Walther (15) sealed thin films of asphalt and tar inside "U-viol" (ultraviolet transmitting) glass tubes. The films used by Von Walther were under different atmospheric conditions in the tubes. Von Walther indicated no change in luster, weight, or per cent insoluble in benzene for the samples that were in a vacuum.

From the results of these investigations, it is apparent that both ultraviolet and infrared radiation tend to hasten the hardening of asphalt. However, the way in which it is accomplished by the two types of radiation is quite different. Infrared radiation penetrates and raises the temperature of the material below the surface. This raised temperature leads to increased oxidation and volatilization, and consequent hardening of the asphalt.

Ultraviolet radiation, on the other hand, can cause or accelerate reactions only in the upper surface of a pavement, roofing surface, or asphaltic paint. Further deterioration of the material depends upon whether the reaction will progress beyond the upper surface. The surface of asphalt pavements is usually composed of selected and graded mineral aggregate particles surrounded by thin films of the asphalt cement. Much of this aggregate may be quartz, which is transparent to ultraviolet radiation.

It is therefore possible for ultraviolet radiation to cause progressive damage to the pavement by slowly irradiating and hardening the asphalt beneath the surface particles of quartz. Eventually, these particles would loosen and be swept away, exposing new surfaces. The purpose of this investigation was to determine whether this deterioration may occur in the absence of oxygen, as should be the existing condition beneath a particular quartz particle on the surface of a pavement.

LABORATORY INVESTIGATION

Two vacuum distilled asphalts were used in the tests, a 90–100 penetration grade (asphalt A) and a 60–70 penetration grade (asphalt B). Asphalt A is specified by New York state for use in hot mix, and asphalt B is primarily used in the city of New York for hot-mix sheet asphalt pavements.

Thin films of these asphalts were prepared by placing a small amount on one of two warm 3 by 1-in. microscope slides. The other slide was then placed on top and pressed to distribute the asphalt evenly. Uniformity of films thus constructed was checked by viewing through a strong light. The thickness of the films was computed by weighing the slides before and after to 1 mg with an analytical balance. The time interval and temperature were recorded and kept as constant as possible to prevent hardening due to the forming process from entering the final results. Film thicknesses ranged from 50 to 70 μ, and the forming time from 3 min 30 sec to 4 min at an average temperature of 70 C.

The ultraviolet radiation was furnished by a 400-w mercury vapor street lamp, having 19.41 w or approximately 23 per cent of the total output between 340 and 400 mμ. This is the portion of the ultraviolet spectrum that some investigators believe to be the most detrimental to asphalts.[3] The transmission characteristics of the microscope slides were investigated, and it was found that the glass was opaque to ultraviolet radiation shorter than 290 mμ, thus stopping all but a very small amount of radiation between 290 and 340 mμ. From 340 mμ to 400 mμ, transmittance increased from 85 to 91 per cent. The visible light (wavelength longer than 400 mμ) emitted by the lamp was assumed to have no effect on the results. This assumption was based on a study of the literature, and not on any conclusive experimental evidence.

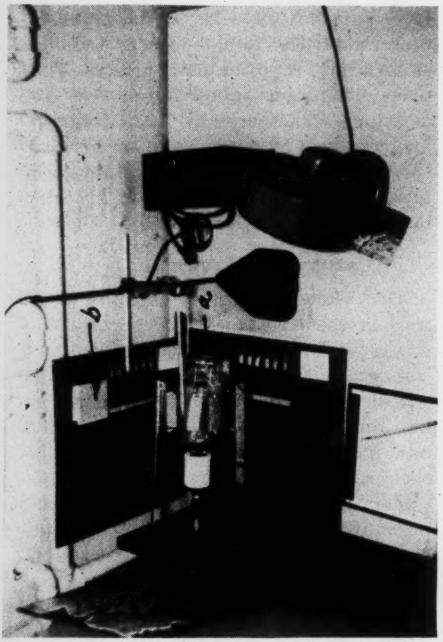

FIG. 1.—Exposure Apparatus.

The films of asphalt were exposed in the rack shown in Fig. 1. The arrow, *a*, points to the bulb which is partially obscured by the curved Plexiglas rack holding samples. Three separate groups of slides are being exposed; the two Masonite boards hold samples 9 in. from the center of the horizontal lamp, while the Plexiglas sample holder is at a distance of 4 in. from the center of the lamp. A wooden shield protecting the control slides is visible on the vertical board at *b*. The control slides for the group held by the Plexiglas were wrapped in lead foil and held between the Plexiglas and the exposed slides. The laboratory stand holds the thermometers. The temperature log, fan, and tranformer are also visible.

At the end of the exposure period, the slides of a particular group were removed, warmed, and slipped apart. Next, the asphalt was scraped with a razor blade from each pair of exposure slides and transferred to a pair of viscosity plates used in the testing instrument. The viscosity plates were then pressed together to form a film, and the homogeneity of the film was checked by viewing in a strong light. The plates were allowed to cool 1 hr before being cleaned, dried, and weighed to 0.1 mg. The weight of the asphalt between the plates was computed by subtracting the original weight of the plates, and the film thickness then computed.

The sliding plate microviscometer, introduced by the Shell Development Co. and described in recent papers (16,17) was used to measure viscosity. This instrument, applies by means of a beam balance, a known load or shearing stress to a thin film of asphalt between two glass plates, 20 by 30 by 6 mm thick. Movement of the glass is followed by a metric

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[3] Unpublished measurements shown to the author during personal interview with Mr. M. H. Veazey, Director of Research, the R. J. Funkhouser and Co., Inc., Hagerstown, Md., Jan. 11, 1957.

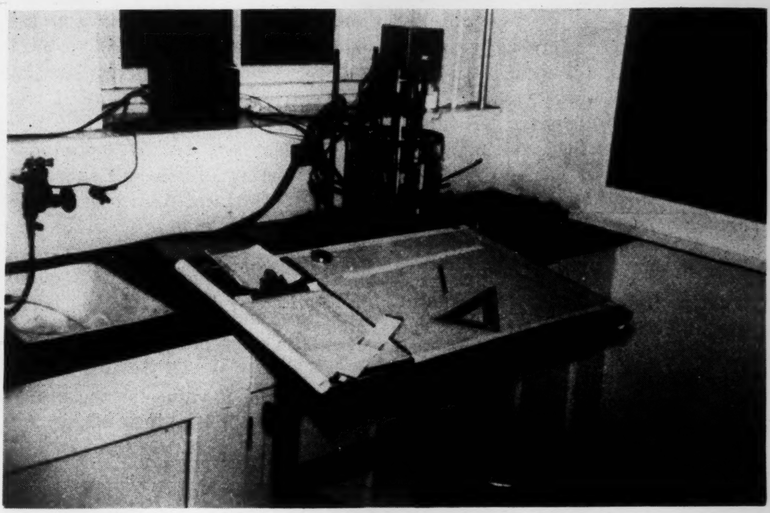

FIG. 2.—Microviscometer, Water Bath, and Movement Chart.

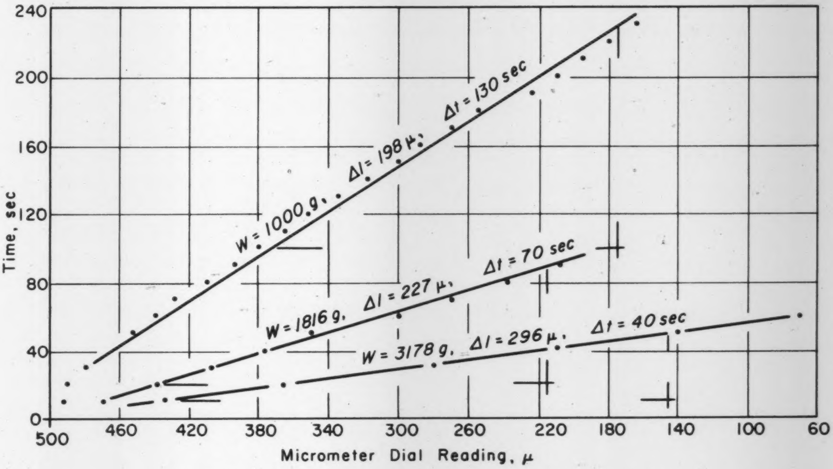

FIG. 3.—Shear Movement *versus* Time for Three Loads.

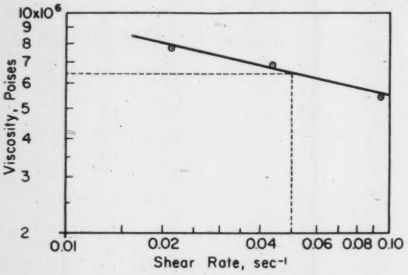

FIG. 4.—Log Shear Rate *versus* Log Viscosity.

micrometer driven by a servo motor. Movement of the glass plates is measured directly by reading the micrometer as a function of time.

TABLE II.—VISCOSITY OF BULK SAMPLES.

Sample	Number of Observations, n	$\bar{x}$, poises	s, poises	Per Cent Standard Deviation
Asphalt B, taken cold from top surface of bulk sample	10	4.922×10^6	1.029×10^6	22.2
Asphalt B, 2 in. below surface, 50 g, stirred, warmed to 100 C	6	3.673	0.133	3.6
Asphalt A, 2 in. below surface, 50 g, stirred, warmed to 100 C	6	1.057	0.0582	5.5

Figure 2 shows viscosity plates in water bath (arrow), with temperature at 77 ± 0.1 F. The plates were allowed to come to this temperature, loads were applied, and the movement in terms of microns *versus* time plotted directly on the table in the foreground.

Figure 3 is an example of the time *versus* movement graphs which were plotted for each viscosity test. Because most asphalts are non-Newtonian (viscosity varies with shear rate), each sample was tested at three different shear rates. For each shear rate, the dial reading was plotted every 10 sec and a straight line fitted by eye through those points. The total movement, Δl, for a given time interval, Δt, was read from this line.

For a fluid between flat plates, it can be shown that the shearing stress is proportional to the rate of shearing strain, and the viscosity constant, η, is then equal to shear stress divided by shear rate:

$$\eta_{(\text{poises})} = \frac{F/A}{V/R}$$

where:

F = force in dynes,
A = area of plates in sq cm,
V = velocity, $\Delta l/\Delta t$, of plates, in μ per sec, and
R = thickness of asphalt film in μ.

Using the R value for the film tested in Fig. 3, three values of viscosity at three different shear rates are computed. These values are plotted as the log viscosity *versus* shear rate (Fig. 4). (This is a straight line for most asphalts.) The necessity for specifying shear rate when discussing the viscosity of a non-Newtonian asphalt is illustrated by the slope of the line in Fig. 4. The shear rate used to compare viscosities in this report was 0.05 sec^{-1}; therefore, the viscosity of this particular film would be reported by interpolation to be 6.5×10^6 poises at 77 F.

In order to determine the extent of hardening caused by heating the asphalt during film construction, the original viscosity of the asphalts as received from the refinery was checked. The results of these measurements, expressed as arithmetic means, $\bar{x}$, and estimated standard deviations, s[4] together with the methods of taking asphalts from sample cans, are summarized in Table II.

Table II illustrates the importance of securing representative samples from a bulk container. In the first instance, a glass rod was forced into the surface of the asphalt, and the small amount which adhered to the rod was transferred to the viscosity plates. This was repeated at random ten times. The excessive standard deviation of this set was probably due to surface hardening. (The asphalt had been stored undisturbed at room temperature for 5 months.) Much better results were obtained in the second and third sets of observations. In each of these sets, approximately 50 g was cut from 2 in. below the surface and transferred to another container, warmed to 100 C in a water bath, stirred, cooled, and then tested.

TABLE III.—VISCOSITIES AFTER 252 HR IN TEST RACK.

Group and Asphalt	Average Test Temperature, deg Cent	Exposure Condition[a]	Number of Observations, n	Mean Viscosity, $\bar{x}$	Estimated Standard Deviation, s	Per Cent Standard Deviation
1-A..........	30.2	Shielded	5	1.46×10^6	0.106×10^6	7.3
1-A..........	30.2	Exposed	5	1.57	0.165	10.5
2-B..........	29.7	Shielded	5	5.67	0.101	1.78
2-B..........	29.7	Exposed	5	6.45	0.278	4.32
3-A..........	41.7	Shielded	5	1.89	0.394	20.8
3-A..........	41.7	Exposed	5	2.50	0.705	28.2

[a] Lamp to film distance was 9 in. except in group 3-A which was 4 in.

TEST RESULTS

The experimental work covered by this report is represented in large part by three separate groups of viscosity data. The procedure for obtaining each was:

1. Constructing and sealing ten films of the same asphalt between microscope slides.
2. Exposing five films to the action of ultraviolet light and shielding the remaining five.
3. Testing the viscosity of all films after a period of 252 hr.
4. Computing the average viscosity for the five exposed films and comparing it to the average viscosity of the five shielded films.

The three groups with test conditions and final viscosities are listed in Table III.

To determine whether the increase in viscosity of the exposed films over the shielded (Table III) is significant in relation to the experimental variation, a Student t test was applied.[4] The assumptions made in order to apply this test were: (1) the underlying population is normally distributed, and (2) similar construction methods and times permit the standard deviation data to be pooled.

The hypotheses were:

$$H_0\colon \bar{x}_{\text{exposed}} = \bar{x}_{\text{shielded}}$$

$$H_1\colon \bar{x}_{\text{exposed}} > \bar{x}_{\text{shielded}}$$

The estimated standard deviation data was then pooled:

$$s_p^2 = \frac{\sum_1^{n_1} (x_1 - \bar{x})^2 + \sum_1^{n_2} (y_1 - \bar{y})^2}{n_1 + n_2 - 2}$$

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[4] The Student t test is a statistical method of making a statement about the relative magnitude between two arithmetic means ($\bar{x}$ (exposed) and $\bar{x}$ (shielded)) which are computed from different sets of data.

And the value of $t_{n_1+n_2-2}$ computed:

$$t_8 = \frac{(\bar{x}_{\text{exposed}} - \bar{x}_{\text{shielded}})}{s_p \sqrt{\frac{1}{n_1} + \frac{1}{n_2}}}$$

From a table of t distributions, the probability of obtaining a deviation (t_8 value) numerically greater than 2.306 is 5 per cent. Therefore, if the computed value of t_8 is larger than 2.306 the first hypothesis, H_0, must be rejected and the second, H_1, assumed true. If the computed value of t_8 is less than 2.306, then the difference between the means is not great enough (at the 95 per cent probability level) to make any statement about their magnitude.

The computed values of t_8 were:

Group 1-A	1.36
Group 2-B	5.93
Group 3-A	1.69

It is evident, then, that only group 2-B passed the test and only for this group is the increase statistically significant. Though both groups 1-A and 3-A show a mean increase, it is not statistically significant because of the large standard deviations. This was probably due in group 1-A to poor technique, both in film formation and testing. The exceedingly large standard deviation of group 3-A was caused indirectly by high test temperatures and resulting loss of asphalt during the exposure period. The amount remaining was insufficient for proper testing in the microviscometer.[5]

CONCLUSIONS

It is generally accepted by most asphalt technologists that hardening of asphalts is a complex phenomeon occurring through the mechanisms of oxidation, volatilization, thixotropy, syneresis, and polymerization. Radiant energy also helps the process along; the infrared portion raises the temperature and promotes volatilization while the ultraviolet accelerates the oxidation of the asphalt.

The results of this investigation, with limited data, indicate that ultraviolet radiation longer than 300 mμ may also harden asphalt even when oxygen is not present. The increase in the viscosity of exposed films of asphalt as compared to control films which were shielded is attributed to the absorption of ultraviolet energy by the asphalt.

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[5] For best results, film thickness in the microviscometer should be in the range of 60 to 100 μ.

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SUMMARY OF PROCEEDINGS OF THE SYMPOSIUM ON EFFECT OF WATER ON BITUMINOUS PAVING MIXTURES

In light of the tremendous national highway program underway throughout the nation, it is timely to make available a group of papers discussing the factors which play a significant part in the behavior of bituminous paving mixtures when exposed to the stripping action of water.

This Symposium on Effect of Water on Bituminous Paving Mixtures was sponsored by ASTM Committee D-4 on Road and Paving Materials. The papers and discussion in this Symposium were presented at the Nineteenth Session of the Sixty-first Annual Meeting of the Society held in Boston, Mass., June 24, 1958, under the chairmanship of C. E. Proudley, North Carolina State Highway and Public Works Commissioner, Chairman of the Special Subcommittee.

A paper by A. B. Brown, J. W. Sparks, and G. E. Marsh entitled "Objective Appraisal of Stripping of Asphalt from Aggregate" presented at the Fifteenth Session was included for publication in this volume because it rounds out the rest of the Symposium.

The papers included in the Symposium are:

Introduction—C. E. Proudley
Field Observations of the Behavior of Bituminous Pavements as Influenced by Moisture—W. K. Parr
Relationship of Aggregate Characteristics to the Effect of Water on Bituminous Paving Mixtures—J. M. Rice
Laboratory and Field Studies of Asphalt Paving Mixtures—E. W. Klinger and J. C. Roediger
Laboratory Study of Anti-Stripping Additives for Bituminous Materials—P. F. Critz
Objective Appraisal of Stripping of Asphalt from Aggregate—A. B. Brown, J. W. Sparks, and G. E. Marsh
Methods of Testing for Water Resistance of Bituminous Paving Mixtures—W. H. Goetz

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THE CONSTANT VOLUME CONDITION AT SMALL PLASTIC STRAINS

By J. D. Lubahn[1]

Synopsis

In tension tests on copper and on an aluminum alloy, using mechanical strain gages, it was found that the constant volume condition applies to small plastic strains. That is, the sum of the offset plastic strains in the three principal directions is zero in the strain range up to 1 per cent. Reanalysis of previous data from the literature confirmed this conclusion.

For strains greater than about 0.002, SR-4 gages gave strain values significantly lower than those from mechanical gages.

The conclusions from this investigation depend heavily upon the accuracy of difficult measurements. Sufficient accuracy probably was attained, but additional work, with still greater accuracy, might be desirable.

Definition of Symbols

e = conventional strain,
ϵ = logarithmic strain,
e'_p or ϵ'_p = permanent strain after load removal,
e_p or ϵ_p = offset from elastic line,
e_z or ϵ_z = strain in direction of loading,
e_x , e_y , ϵ_x , or ϵ_y = strain in two transverse directions perpendicular to each other,
$\epsilon_{tp\ \mathrm{av}}$ = average of plastic strains in x and y directions,
e_m or ϵ_m = measured strain,
e_c or ϵ_c = strain that has been corrected for transverse strain,
e_l or ϵ_l = strain in the measuring direction,
ϵ_t = strain transverse to the measuring or loading direction,
μ_0 = elastic value of Poisson's ratio, and
K = experimental constant used in correcting for the strain transverse to the measuring direction

It has been recognized for many years that the volume of a metal is not appreciably affected by forming operations. In rolling sheet, for example, one observes that, with essentially no change in width, a 50 per cent decrease in thickness results in doubling the length.

Such observations apply, of course, to dimensions measured after the forming process. In other words, the dimensional relationships are expressed in terms of the permanent strains remaining after removal of the load. The principle that the volume is not affected by permanent strain can be expressed either as:

$$(1 + e'_{xp})(1 + e'_{yp})(1 + e'_{zp}) = 1, \ldots (1)$$

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or as:

$$\epsilon'_{xp} + \epsilon'_{yp} + \epsilon'_{zp} = 0 \ldots\ldots\ldots (2)$$

where x, y, and z are any three mutually perpendicular directions and e'_p and ϵ'_p are conventional strain and logarithmic strain defined as:

$$e'_p = \frac{\text{permanent change in length}}{\text{initial length}}, \ldots (3)$$

and:

$$\epsilon'_p = \ln \left(\frac{\text{length after plastic flow and removal of the load}}{\text{initial length}} \right) \ldots (4)$$

Equations 1 and 2 are very useful in dealing with metal forming problems such as rolling, drawing, upsetting, extrusion, etc., and their use is described in numerous technical papers, as well as in a reference book on this subject by Sachs and Van Horn (**1**).[2]

For the large plastic strains involved in such forming operations, Eqs 1 and 2 are well established, by wide experience within the forming industry, as being sufficiently correct for practical purposes. Apparently Eqs 1 and 2 are also valid for small plastic strains. Statements to this effect, but without supporting data, have been made by many investigators, such as Taylor and Quinney (**6**), on the basis of density measurements before and after deformation. Many of these density measurements are not accurate enough to insure a high degree of conformity to Eqs 1 and 2 at a low level of plastic strain, while other measurements are accurate enough to indicate a very high degree of conformity. Let us suppose, for example, that one wished to know the ratio of a transverse strain to the longitudinal strain for conditions where the two mutually perpendicular transverse strains were equal to each other. This ratio would be equal to $-\frac{1}{2}$, according to Eq 2, if the strains are expressed as logarithmic strains. In Goodman and Russell's work (**4**), then, an accuracy of density measurements of about 0.02 per cent only serves to show that the transverse-to-longitudinal strain ratio is within 10 per cent of $-\frac{1}{2}$ at a longitudinal strain of 0.002, while Stowell and Pride's (**3**) accuracy of density measurements of about 0.0014 per cent serves to show that this strain ratio is within 1 per cent of $-\frac{1}{2}$ at a longitudinal strain of 0.002. Smith (**7**) reported volume changes for different metals ranging from -0.07 to $+0.09$ per cent.

Often it is necessary to use an equation like Eqs 1 or 2 to solve the plasticity problems that arise in connection with design of load-carrying structures that are permitted to undergo small amounts of plastic flow (see reference (**19**), for example). In such design considerations, the plastic strains involved are usually considerably less than 1 per cent, and the problem invariably requires knowledge of the plastic strains under load, rather than after removal of the load. It has been widely assumed in solving these plasticity problems that equations similar to Eqs 1 and 2 apply to the plastic strains that exist under an applied load, and that this applicability extends to very small values of plastic strain. However, the experimental data which are available to justify this assumption are very scanty and confused. These data will be reviewed and interpreted, and then additional data bearing on the question will be presented.

In dealing with data taken while the metal is under load, it is necessary to define plastic strain in a different way than in Eqs 3 and 4. In plasticity problems for design engineering purposes, what is needed is a relationship between the principal plastic strains defined as the difference between the total strain and

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[2] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 1344.

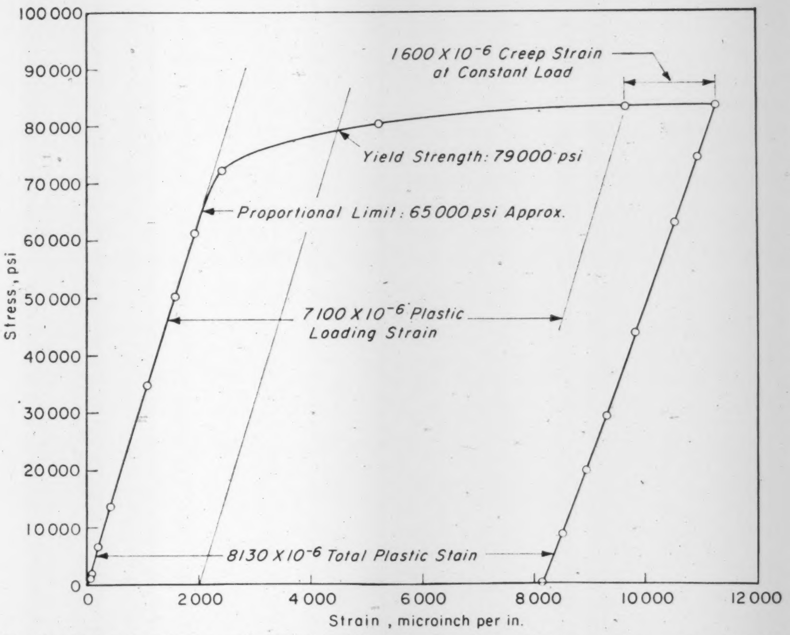

FIG. 1.—Stress-Strain Curve for Type 403 Stainless Steel Quenched and Tempered to 80,000 p Yield Strength.

After slow loading at constant strain rate, the specimen was allowed to creep at maximum load for 1 hr before slow unloading. From reference (**5**).

the elastic strain, or the offset from the elastic line. In these plasticity problems, the total strains in various directions are related to each other by the compatibility conditions and the elastic strains are related to the stresses by Hooke's law. Plastic strain defined by Eqs 3 and 4 is not the same as the offset from the elastic line, because of the characteristic curvature of the unloading line after plastic flow has occurred. In Fig. 1, for example, the offset from the elastic line is 0.00870, while the permanent strain is only 0.00813. The fact that the materials of this investigation showed similar behavior is shown by Fig. 16. In this paper, symbols e'_p and ϵ'_p, as defined in Eqs 3 and 4, are used to indicate the permanent strains remaining after unloading; and the symbols e_p and ϵ_p are used to indicate plastic strains as defined by the offset from the elastic line, or the "offset plastic strains:"

$$e_p = \frac{\text{length at load-initial length}}{\text{initial length}} - \frac{\text{stress}}{\text{Young's modulus}} \quad \text{..(5)}$$

$$\epsilon_p = \ln\left(\frac{\text{length at load}}{\text{initial length}}\right) - \frac{\text{stress}}{\text{Young's modulus}} \quad \text{..(6)}$$

With this notation, equations similar to Eqs 1 and 2, can be written applying to the offset plastic strains:

$$(1 + e_{xp})(1 + e_{yp})(1 + e_{zp}) = 1 \quad \text{...(7)}$$

$$\epsilon_{xp} + \epsilon_{yp} + \epsilon_{zp} = 0 \quad \text{.........(8)}$$

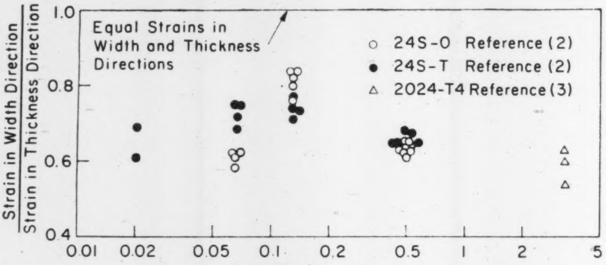

FIG. 2.—Ratio of Transverse Strains for Tension and Compression Specimens Cut in the Rolling Direction from Aluminum Alloy Sheet and Plate.

Data on 2024-T4 are for compression of square specimens at 0.004 to 0.01 plastic strain. Data on 24S are for tension of wide and narrow strips at about 0.14 plastic strain.

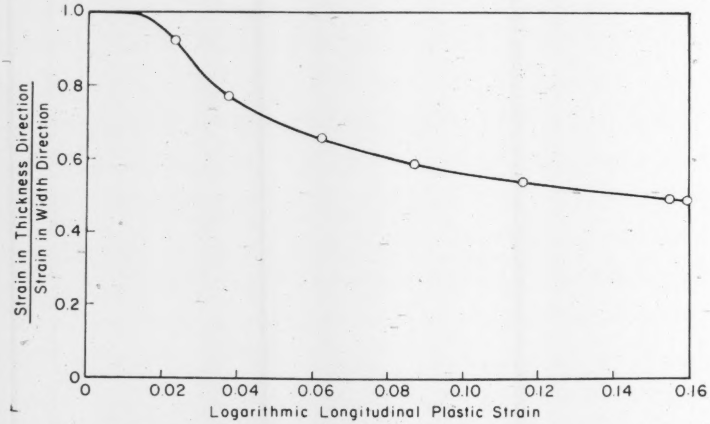

FIG. 3.—Ratio of Transverse Plastic Strains for Tensile Specimen Cut in the Rolling Direction from FS-1h Magnesium Alloy Sheet.

From the data of reference (**4**).

Much of the data in the literature on tension and compression tests which can be used to determine the validity of Eqs 7 and 8 is presented in the form of ratios of transverse to longitudinal strain, or "Poisson's ratio." In forming this ratio, it is customary to use the total conventional strain, including both the elastic and plastic portions. It is often stated that Poisson's ratio changes gradually from its value in the vicinity of 0.3 in the elastic region to a value which approaches $\frac{1}{2}$ at large plastic strains. It must be recognized, however, that this ratio will *not* approach $\frac{1}{2}$ unless several conditions are satisfied:

1. The two transverse strains are equal.

2. The deformation is expressed in terms of logarithmic strain rather than conventional strain,[3] and

3. Equations 7 and 8 are valid.

If the specimen is made from drawn rod, the transverse strains will be very nearly equal, because of axial symmetry. Under these conditions Eq 8 reduces to:

$$\frac{\epsilon_{xp}}{\epsilon_{zp}} = \frac{\epsilon_{yp}}{\epsilon_{zp}} = -\frac{1}{2} \qquad (9)$$

In other words, the criterion of conformity to the constant volume condition under conditions of axial symmetry is that the ratio of either transverse logarithmic plastic strain to the longitudinal logarithmic plastic strain is $-\frac{1}{2}$. In a sense, this is similar to saying that Poisson's ratio is $\frac{1}{2}$ except that we are dealing with the plastic portion of the strain and expressing it as logarithmic strain, instead of dealing with total strain (including the elastic portion) and expressing it as conventional strain.

If a rod- or strip-shaped specimen is cut in the rolling direction from sheet or plate, the transverse strains will usually be unequal in the width and thickness directions of the sheet. The two transverse strains may be unequal from the start and remain in approximately constant ratio, as in the aluminum alloys of Fig. 2, or the strains may become unequal as the deformation proceeds, as in the magnesium alloy of Fig. 3. Under these conditions, Eq 8 can be written in the form:

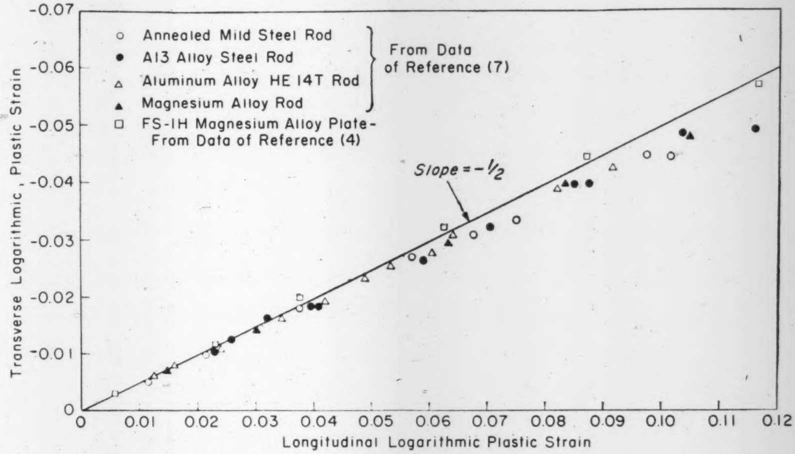

FIG. 4.—Results of Tension Tests on Several Rod and Plate Materials Using Mechanical-Type Strain Gages.

For the plate tests, the ordinate values are the average of the strains in the width and thickness directions.

$$\frac{\epsilon_{tp\,\mathrm{av}}}{\epsilon_{zp}} = -\frac{1}{2}, \qquad (10)$$

where $\epsilon_{tp\,\mathrm{av}}$ is the average transverse logarithmic plastic strain:

$$\epsilon_{tp\,\mathrm{av}} = \frac{\epsilon_{xp} + \epsilon_{yp}}{2} \qquad (11)$$

In other words, the criterion of conformity to the constant volume condition for transversely asymmetric conditions is that the ratio of the average transverse to the longitudinal logarithmic plastic strain is $-\frac{1}{2}$.

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[3] At 100 per cent conventional strain, the cross-section is ½ the original, the diameter is 0.707 times the original, the transverse strain is −29.3 per cent, and Poisson's ratio appears to be 0.293, rather than 0.500.

Equations 7 and 8 are the equations which are in suitable form to be used in plasticity problems where small plastic strain accompanies the elastic strain. Equations 9 and 10 are suitable criteria for judging whether Eqs 7 and 8 are applicable in a tension or compression test. In this report we shall be concerned primarily with the validity of Eqs 7 and 8. The fact that the density measurements described above indicate Eqs 1 and 2 to be valid does not mean that Eqs 7 and 8 are also valid, because of the difference in the plastic strains defined by Eqs 3 and 4 and by Eqs 5 and 6, as illustrated in Fig. 1. However, since plastic strains by the two definitions are not greatly different, we may conclude from the density measurements that Eqs 7 and 8 are at least approximately correct.

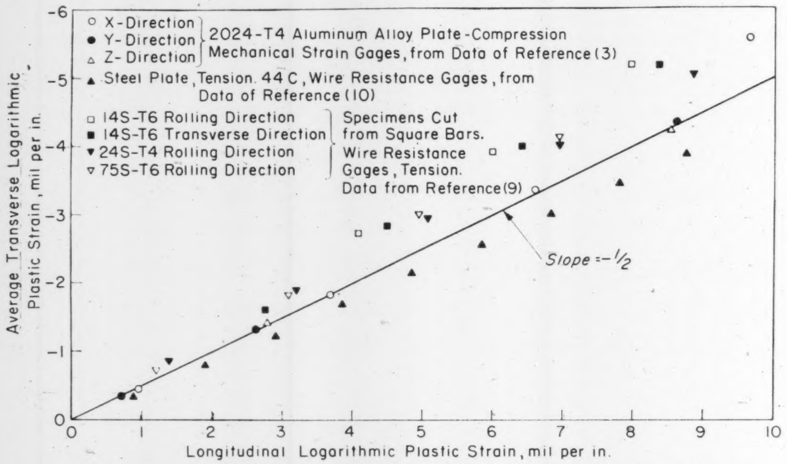

FIG. 5.—Results of Compression and Tension Tests on Several Materials.

Direct evidence of the validity of Eqs 7 and 8 is almost entirely lacking in the technical literature. There are many investigations where longitudinal and transverse strains have been measured, and values of Poisson's ratio have been reported, but these data are not entirely satisfactory for showing the validity of Eqs 7 and 8 for one or more of the following reasons:

1. The plastic strains are not small enough,

2. The strain measurements are not reliable,

3. Only one transverse strain was measured, and the other cannot be assumed to be equal to it, and

4. The data do not apply to a wide enough variety of materials to draw a general conclusion.

Figure 4 shows test results from the literature at comparatively large strains, expressed in a form suitable for comparing with Eqs 9 or 10. Similar results could be obtained for 24S-T aluminum alloy by suitable analysis of Klingler and Sachs' data (8), and for commercially pure titanium by analysis of Goodman and Russell's data (4). Figure 4 shows that Eqs 7 and 8 are very nearly correct in the range of longitudinal logarithmic plastic strain from 1 or 2 per cent to about 10 per cent.

The scattering about a trend line is such, however, that the validity of Eqs 7 and 8 cannot be demonstrated for strain levels less than 1 per cent.

Figure 5 shows test results from the literature at plastic strains less than 1 per cent, expressed in a form suitable for comparison with Eq 10. At these small strains, e and ϵ are substantially equal, and so conventional strains were plotted instead of the logarithmic strains called for by Eqs 7 and 8. The same procedure will be used in reporting results of new tests later in this report.

In Fig. 5, a slope of $-\frac{1}{2}$ represents conformity to Eq 10. The actual experimental slopes range from -0.44 for low-carbon steel to -0.50 and -0.61 for aluminum alloys. Other ferritic steels and stainless steel also have been considered (**3,10**) but conformity to Eq 10 cannot be assured because only one of the two transverse strains was measured. Stang, Greenspan, and Newman (**10**) investigated the steel plate at room temperature, as well as at the low temperature mentioned in Fig. 5, and found behavior (strain ratio of -0.45 at 2 per cent strain) similar to that at low temperature.

The reason for the differing slopes in Fig. 5 is not immediately evident. Apparently the variations in slope do not represent real differences between materials, because considerable difference occurred in two different investigations of the same material (24S-T aluminum alloy). This discrepancy in the data on 24S-T aluminum alloy in Fig. 5 will be considered in more detail later. For the moment, it is only important to note that the data in reference (**9**) were obtained with SR-4 gages, while those in reference (**3**) were obtained with mechanical gages. The SR-4 gage readings in reference (**9**) were corrected for transverse strain, using quite a large factor obtained from a theoretical analysis that is not consistent with the experimental correction factors given by Perry and Lissner (**13**). In reference (**10**), SR-4 gage measurements also were used,[4] but without any correction for transverse strain.

Information pertaining to the validity of Eqs 1, 2, 7, and 8, as reported in the technical literature, may now be summarized as follows:

1. Density measurements show fairly well that Eqs 1 and 2 are valid, but these density measurements cannot be used to show that Eqs 7 and 8 are valid, and
2. Measurements of strains in various directions show that Eqs 7 and 8 are valid for large strains; the evidence at small strains, of interest in design engineering problems, is conflicting.

The objective of the tests now to be described was to determine whether Eqs 7 and 8 are valid at small plastic strains for a variety of materials.

EQUIPMENT AND EXPERIMENTAL PROCEDURE

In preliminary tests, small specimens were tested, but later larger specimens were used to attain higher accuracy of transverse strain measurements by mechanical methods, by virtue of the longer gage length. For the small specimens, a high degree of concentricity of loading was obtained by using a special fixture described in an earlier paper (**20**). The larger specimens could not be handled by this loading fixture, and so approximate concentricity was obtained by trial-and-error shimming under the nuts holding each end of the specimen. It was found that moderate amounts of eccentricity were not as troublesome as other difficulties to be described. The detrimental effects of eccentricity were largely avoided by averaging diametrically opposite gages, or by measuring both transverse and axial strain on the same side of the specimen.

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[4] According to "Methods of Test" in reference (**10**), both SR-4 gages and Tuckerman gages were used. However, it seems likely that in these tests on $1\frac{1}{8}$ in. round bars, Tuckerman gages (with 1 in. gage length) were not used for measuring the transverse strains.

It was considered desirable to measure strain by two independent methods, so all specimens were provided with SR-4 wire resistance strain gages and also with mechanical gages. In general, the mechanical gages were considered more desirable than the SR-4 gages, because they could be calibrated before and after the test. Such a procedure insures that the test results using mechanical gages are correct within the limits of accuracy of the calibration. Of course, representative SR-4 gages from a given lot of gages are calibrated in advance by the manufacturer, but there is no way to check the calibration after the test, and furthermore it is never the actual gage used in the test that has been calibrated. On the other hand, all the evidence that could be found in the technical literature indicated that wire resistance strain gages are reliable up to the point where wire breakage causes an open circuit.

For continuous loading in one direction, Kammer and Pardue (**11**) and Swainger (**12**), for example, show that the kind of wire used in SR-4 gages (such as constantan) has linear strain-resistance change curves up to large plastic strains.[5] Stang, Greenspan, and Newman (**10**), who measured strains up to about 2 per cent with both SR-4 gages and Tuckerman gages, did not report any discrepancy in the measurements. Perry and Lissner, in their treatise on wire resistance gages (**13**), do not mention any discrepancies in calibration of these gages. In short, at the outset of the investigation there was no reason to doubt the validity of SR-4 strain measurements in the strain range from 0 to 1 per cent.

The SR-4 gages were applied in the manner recommended by the manufacturer. They were always applied to the specimen in pairs, at opposite ends of a diameter. The strains from the two gages were averaged to obtain centerline strain. In all the tests there were two pairs of axial gages and two pairs of tangential gages, so that two values of centerline strain in each direction were available. Differences of 10 per cent between the two values were common, particularly in the tangential direction. The reported values are the averages for the four gages in a given direction.

The SR-4 gage readings were corrected for the strain in the direction transverse to the measuring direction. This correction was negligible for the axial direction, and ranged from 4 to 8 per cent for the transverse direction, depending on the value of Poisson's ratio and the type of gage used (usually A-7 or A-8). The correction was made according to the following equation, derived from reference (**13**):

$$e_c = e_{ml} - K[e_{ml}(\mu_0 - K) + e_{mt}(1 - K\mu_0)]$$

where e_c is the corrected strain in the measuring direction, e_{ml} is the measured strain in the measuring direction, K is a constant determined experimentally (-0.02 for A-8 gages and -0.01 for A-7 gages according to Perry and Lissner (**13**)), μ_0 is the value of Poisson's ratio in the elastic region, and e_{mt} is the measured strain in the direction transverse to the measuring direction.

Because of the large number of measurements, strain, load, and time readings were taken "on-the-fly" under conditions of constant head speed. The readings on the different gages were taken in rotation, and simultaneous values were obtained by interpolation with respect to load or time, whichever resulted in more accuracy by virtue of there being a larger and more easily measured change.

The problem of contact errors in

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[5] For single-strand gages, this linearity also prevailed when the wire was cemented to a test specimen (**12**).

switching from one gage to another was studied in some detail. It was found that knife switches introduced somewhat less error than SR-4 gage switches or thermocouple switches (about 10 microinches per inch). However, these errors were random and did not significantly affect the reliability of the final result unless the total effect being studied was exceedingly small.

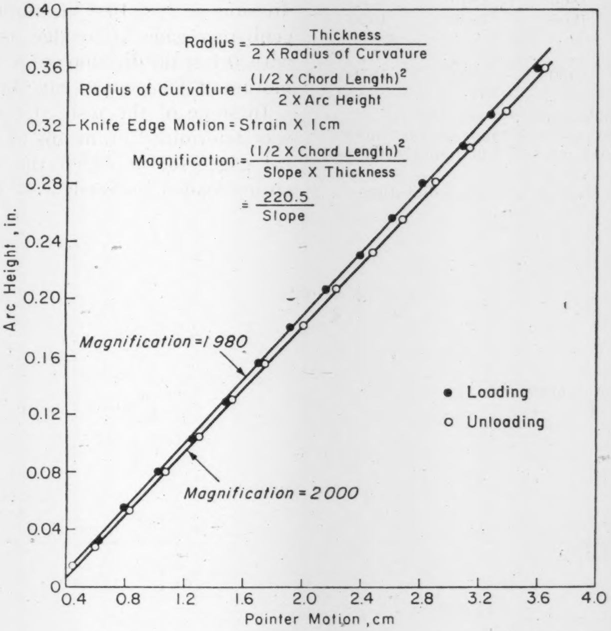

FIG. 6.—Typical Calibration Curve of Huggenberger Extensometer No. 412 in Tensile Loading. Gage length = 1 cm.

In some of the tests the axial strains were measured with diametrically opposite Huggenberger extensometers having a gage length of 1 cm and magnification factor of about 2000. A gage length of 1 cm was used for long travel and low sensitivity, and a gage length of 17.9 cm for short travel and high sensitivity. For a given sensitivity, maximum travel was obtained by periodic resetting of the pointer fulcrum carriage. The load was reduced slightly during these resettings to avoid creep. The Huggenberger extensometers were calibrated against curvature of an elastic beam 0.247 in. thick, using a 0.001-in. dial gage to measure arc height above a 14$\frac{3}{4}$ in. chord. With such an arrangement, full travel of the pointer on the Huggenberger scale corresponded to 0.46 in. of travel on the dial gage, which could be measured easily to an accuracy of 1 per cent. A typical calibration curve is shown in Fig. 6, and a summary of several calibrations on both gages is given in Table I. Table I also gives the sensitivity, reliability, and amount of mechanical take-up.

The calibration uncertainty of 2 per cent is probably due to variations in the clamping force used in applying the

TABLE I.—SUMMARY OF HUGGENBERGER EXTENSOMETER CALIBRATIONS.

Gage No. 412		Gage No. 431	
Upscale Motion	Downscale Motion	Upscale Motion	Downscale Motion
1980	2000	1945	1945
1970	1970	1935	1935
1940	1860	1840	1770
1930	1860	1860	1820
Average: 1955	1920	1895	1870

Calibrating uncertainty:[a] 2 per cent
Scatter of data points: 0.2 mm (pointer motion)
Mechanical take-up: 0.7 mm (pointer motion)

[a] Probable error of a single calibration.

gages. Such variations can cause as much as 5 per cent calibration uncertainty, as discussed by Vose (14). Somewhat better calibration reproducibility can be obtained with Tuckerman gages (15), but Huggenberger gages were preferred as being much easier to read, reliable enough for the purpose, and at least as sensitive as Tuckerman gages (0.1 scale division = 2 × 10^{-6} in. motion of Huggenberger gage knife-edge as compared with 0.1 scale division = 4 × 10^{-6} in. motion of Tuckerman gage knife-edge).

In some of the tests the axial strain was determined by means of a mechanical gage (16) in which the motions of spring-loaded knife-edges at the ends of

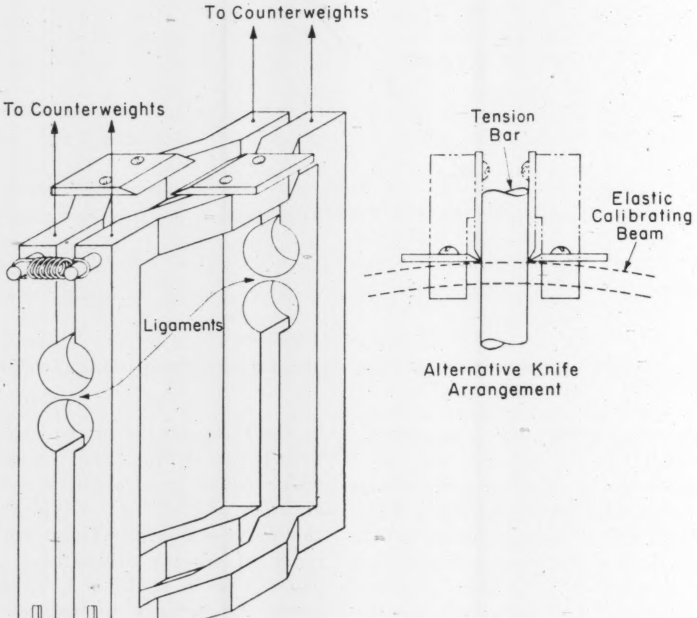
To Counterweights
To Counterweights
Ligaments
Knife-Edge Seats for Huggenberger Gages
Tension Bar
Elastic Calibrating Beam
Alternative Knife Arrangement

FIG. 7.—Scissors Gage for Diameter Measurement (spring not used in calibrating).

the gage length were transferred to ball-bearing carriages by long, flexible rods, and the relative motion of the carriages measured with a microformer-type displacement gage.

Two types of mechanical gages were used for measuring diameter. The older type consisted of two spring-loaded knife-edges moving on guide rods and having a 0.0001-in. dial gage to measure the relative motion of the two knife-edges. The guide rods were lubricated and the spring tensions adjusted to avoid any cocking of the knife-edges on the guide rods. Freedom from sticking was evidenced by smooth motion of the knife-edges as they followed the contraction of the specimen. This gage proved to be sensitive enough to detect plastic strains of about 0.00002.

A new type of diameter gage, Fig. 7, was developed specifically for this investigation. In operation it resembled a pair of scissors with the specimen pinched between the handles and the relative motion of the points of the blades being measured by Huggenberger gages. In principle of operation, this "scissors" gage resembled diameter gages described previously (**17,18**); however Huggenberger gages were used instead of a light beam to measure the motion of the arms. The scissors gage was suspended from its own center of gravity to avoid the action of gravity forces on any of its parts. Elastic ligaments were used as hinges, one at each side of the square frames. It was found in early tests that motion was not transferred reliably through a spring steel ligament clamped at both ends, so

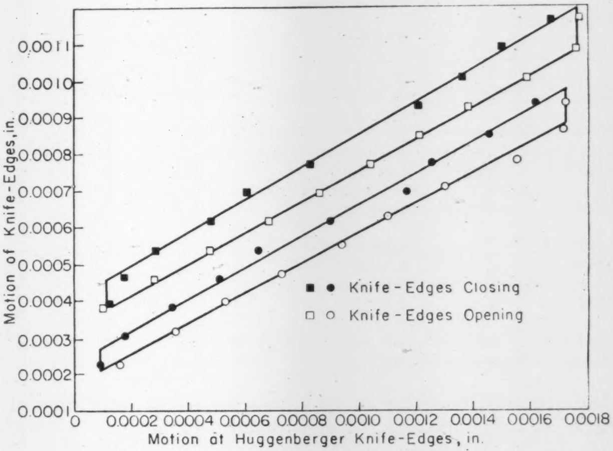

FIG. 8.—Two Typical Scissors Gage Calibrations.

TABLE II.—SUMMARY OF SCISSORS GAGE CALIBRATIONS.

	Magnification Ratio	
	Knife Edges Closing	Knife Edges Opening
	2.27	2.44
	2.22	2.40
	2.20	2.53
	2.27	2.29
	2.18	2.49
Average	2.23	2.43

Calibration uncertainty: 2 per cent
Scatter of data points: 0.00002 in. at knife edges
Mechanical take-up: <0.001 in.

the ligaments had to be made integral with the rotating members by leaving a thin wall between two drilled holes, as shown in Fig. 7.

Readings from the two Huggenberger gages at opposite sides of the square frames were averaged to obtain the motion at a point directly opposite the measuring knife edges. By this means, any possible error from rotation of the frames about a vertical axis was avoided. The scissors gage had a nominal multiplication ratio of two by virtue of its own lever ratio, and a nominal factor of 2000 due to the lever system of the Huggenberger gages. The apparent magnification in calibration experiments was somewhat larger than nominal, presumably because of the deflection characteristics of the ligaments. A typical calibration curve is shown in Fig. 8, and Table II summarizes the results of several independent calibrations. The scissors gage was calibrated by measuring surface strain in a 3.44-in. gage length on an elastically bent beam 0.247 in. thick, and calculating this surface strain from curvature, based on arc-height measurements, in exactly the same way as in the Huggenberger calibrations. The scissors gage was adapted to surface strain measurement by replacing its horizontal knives with vertical knives with the edges at the same distance from the ligaments, as shown by the dotted lines in Fig. 7.

All the mechanical gages exhibited lost motion. This was small but significant for the precision being sought here because of the loss of effective gage travel. In most cases this mechanical take-up could be largely eliminated before the test by springing the parts elastically and then releasing them in such a way that motion in the desired direction had already commenced before the test.

TABLE III.—ELASTIC BEHAVIOR OF 24S-T ALUMINUM ALLOY.

	Axial Strain at 32,000 psi, Elastic Limit		Tangential Strain at 32,000 psi, Elastic Limit		Modulus of Elasticity		Poisson's Ratio	
	SR-4 Gages	Huggenberger Gages	SR-4 Gages	Diameter Gage	SR-4 Gages	Huggenberger Gages	SR-4 Gages	Mechanical Gages
	0.00275 0.00290 [a]	0.00304 0.00317 [a]	0.00087 0.00089 [a]	0.00099	11.4×10^6	10.3×10^6	0.311	0.319
Average	0.00282	0.00310	0.00088					
	0.00279 0.00288 [a]		0.00087 0.00090 [a]		11.3×10^6		0.309	
Average	0.00284		0.000885					

[a] Bracket indicates a pair of diametrically opposite gages. Difference is a measure of the eccentricity in the plane of the pair of gages.

TEST RESULTS

One bar of 24S-T aluminum alloy and two bars of annealed OFHC copper were tested. The 24S-T aluminum alloy specimen was instrumented with two pairs of axial SR-4 gages, two pairs of tangential SR-4 gages, a pair of Huggenberger gages for axial strain, and a dial-indicator-type diameter gage. The results are shown in Table III and in Figs. 9 and 10.

Table III shows that the SR-4 gages gave 10 per cent smaller elastic strains than the mechanical gages. This disagreement increased as plastic strain proceeded, reaching as much as 28 per cent in the case of the tangential strains (Fig.

9). According to the mechanical gages, the elastic constants of $E = 10.3 \times 10^6$ psi and $\mu_0 = 0.32$ agreed closely with values reported (10) in the literature ($E =$ 10.0 to 10.7×10^6 psi and $\mu_0 = 0.310$ to 0.331).

Figure 10 shows the relationship between tangential and axial plastic strain. According to the mechanical gages, the ratio of the two strains is very nearly $-\frac{1}{2}$, which indicates that the constant volume condition, Eqs 7 and 8, does apply. According to the SR-4 gages, however, the ratio of tangential to axial plastic strain is much smaller, as was found by the author in earlier tests on heat-treated steel and by Stang, Greenspan, and Newman (10), Fig. 5.

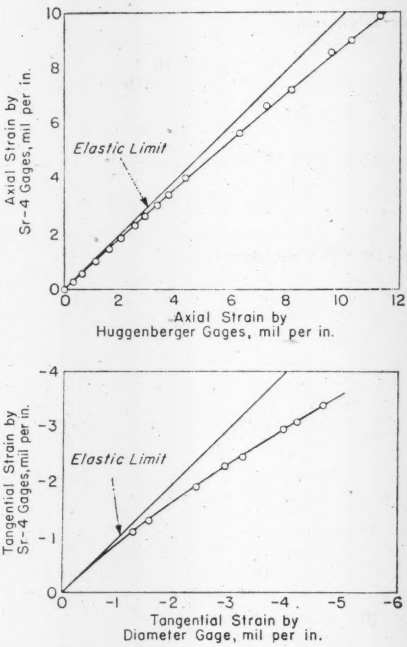

FIG. 9.—Comparison of Strain Measurements by Mechanical and Electrical Gages. 1-in. diameter 24S-T aluminum alloy bar.

Two tests, using both mechanical and SR-4 gages, were made on 1-in. diameter bars of OFHC copper. These were rough machined in the cold-drawn condition

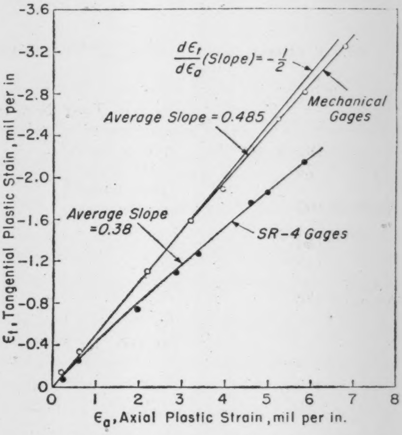

FIG. 10.—Plastic Behavior of 24S-T Aluminum Alloy Bar.

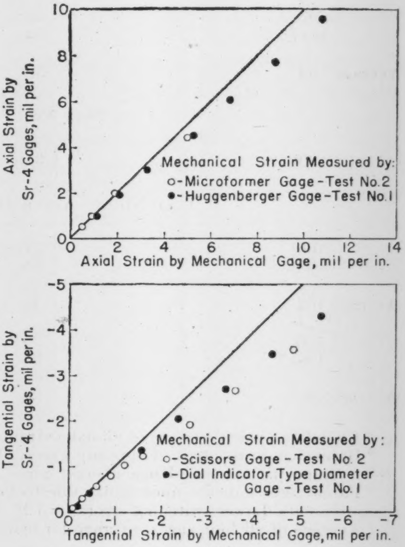

FIG. 11.—Comparison of Strain Measurements by Electrical and Mechanical Methods. OFHC copper.

TABLE IV.—ELASTIC BEHAVIOR OF ANNEALED OFHC COPPER.

Strain at Elastic Limit (1800 psi)				Elasticity Constants			
Axial		Tangential		Modulus		Poisson's Ratio[a]	
SR-4 Gages	Mechanical Gages	SR-4 Gages	Mechanical Gages	SR-4 Gages	Mechanical Gages	SR-4 Gages	Mechanical Gages
TEST NO. 1—AS ANNEALED							
137 × 10^{-6}[b] 73	110 × 10^{-6}[c] (17.9 cm gage length)			(17 × 10^6)[d]	16.3 × 10^6		
Average 105							
114 118				(15)			
Average 116							
TEST NO. 1—AFTER 40 × 10^{-6} PLASTIC STRAIN							
114 60	125 79	40 × 10^{-6} 22		(21)	(17.7)	(0.32)	
Average 87	102 (1 cm gage length)	31					
97 101		31 33		(18)		(0.34)	
Average 99		32					
TEST NO. 2—AS ANNEALED							
99[e]		31[e]		18		(0.31)	
TEST NO. 2—AFTER 10 × 10^{-6} PLASTIC STRAIN							
106 98		31 35	39 × 10^{-6}[e]	18		(0.33)	0.38
Average 102		33					
100 106		35 35		17.5		(0.36)	
Average 103		35					

[a] Calculated using average of all axial strain measurements.

[b] Bracket indicates pair of opposite gages. Difference indicates initial eccentricity.

[c] Average of 2 opposite Huggenberger gages as axial gages or used with scissors gage.

[d] Parentheses indicate uncertainty due to very small total effects compared with sensitivity of measurements. Lower switching errors (± 3 × 10^{-6}) give higher sensitivity on second test.

[e] Average of all four gages; average for loading and unloading after 10 × 10^{-6} plastic strain.

and annealed at 400 C for 2 hr before final machining. With copper, the values of small plastic strains can be determined more accurately than in 24S-T aluminum alloy because the elastic strains are much smaller.

In the first test on copper, the specimen was provided with two pairs of axial SR-4 gages and two pairs of tangential SR-4 gages; also axial strain was measured with the Huggenberger gages and transverse strain was measured with a dial-indicator-type diameter gage. In the second test on copper, the same array of SR-4 gages was used; and in addition axial strains were measured with the microformer-type displacement gage, and the Huggenberger gages were used on the scissors gage in the measurement of tangential strain. Figure 11 and Table IV show that the mechanical and SR-4 gages agree moderately well in the elastic region[6] and up to about 0.001 or 0.002 total strain, but then the same type of deviation observed earlier sets in. Consequently the results of the SR-4 gage measurements were considered to apply only up to about 0.002 total axial strain or 0.001 total tangential strain.

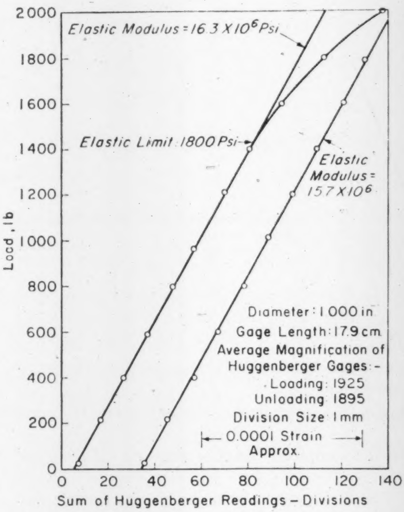

FIG. 12.—First Tensile Test of Annealed OFHC Copper.

Figure 15 shows the results of the two tests on copper, expressed in a form suitable for comparison with Eq 9. The ratio of tangential to axial plastic strain is very close to the ideal value of $-\frac{1}{2}$ in both tests, if strain values from mechanical gage measurements are used. The ratio is also $-\frac{1}{2}$ up to the limit of reliability of the SR-4 gages (0.002 axial strain). The ratio is not far from $-\frac{1}{2}$ for larger SR-4 strains, but this result is fortuitous: it occurs only because the deviations from correct values (Fig. 11) are similar for the axial and tangential directions, thus producing compensating errors. The failure to exhibit a plastic strain ratio of $-\frac{1}{2}$ in Fig. 10 was due to the greater deviation of the tangential strains than of the axial strains in Fig. 9. No explanation could be found for the varying divergencies of the SR-4 gages (particularly tangential gages) in the various tests; in the copper tests, some of the tangential gages were type A-7 and some were type A-8, and in both tests these two types agreed with each other as closely as gages of the same type agreed with each other.

As indicated by Figs. 9 and 11, types A-7 and A-8 SR-4 gages were not found suitable for judging the validity of Eqs 7 and 8. It was found, subsequent to this

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[6] The degree of agreement in the elastic region is difficult to ascertain for this material because of the very small strain at the elastic limit. Only the ultra-high-sensitivity mechanical gages yielded reliable elastic slopes (Figs. 12 and 13); because of switching errors, the SR-4 gages exhibited scatter which was often severe in comparison to the total elastic effect (Fig. 14).

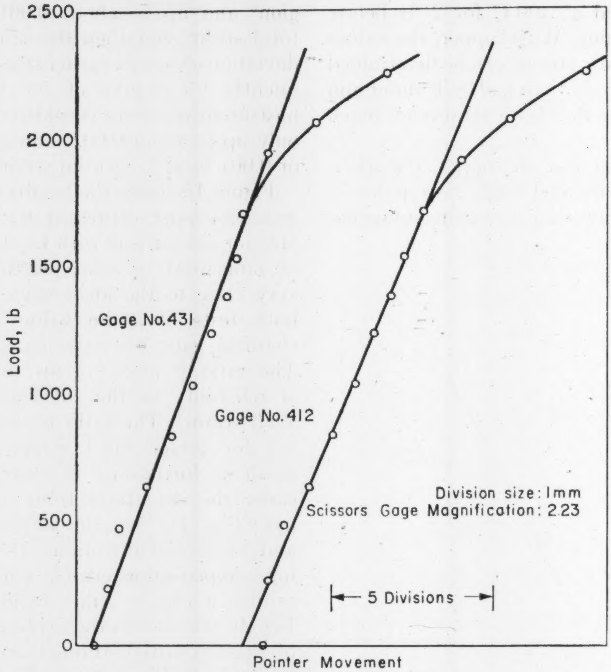

FIG. 13.—Scissors Gage Data for Determining Load-Diameter Curve. Annealed OFHC copper after 40 × 10^{-6} plastic stretching.

Huggenberger gages No. 431 and No. 412 attached at opposite sides of scissors gage frame.

investigation, that similar unreliability had been observed earlier by Campbell (21). For the A-7 gage (*M* in Campbell's work), in particular, he found about 5 per cent error at 0.002 strain and about 10 per cent error at 0.006 strain. These errors are of the same order of magnitude as those depicted in Figs. 9 and 11. If, on the basis of Figs. 9 and 11 and Campbell's work, we disregard the data of references (9) and (10) and the SR-4 gage data of Figs. 10 and 15, we are left only with the data from mechanical gages shown in Figs. 5, 10, and 15. These data indicate that Eqs 7 and 8 are correct for copper and 24S-T aluminum alloy. This conclusion should be considered a pre-

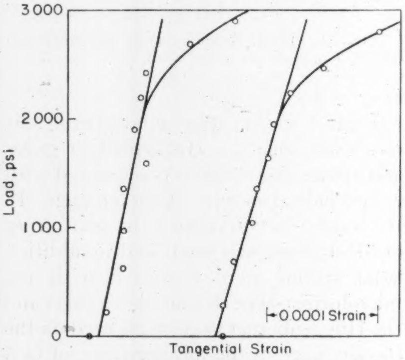

FIG. 14.—Typical Load-Strain Curves Using SR-4 Gages Showing Scatter and Slope Uncertainties. Annealed OFHC copper; 1-in. diameter.

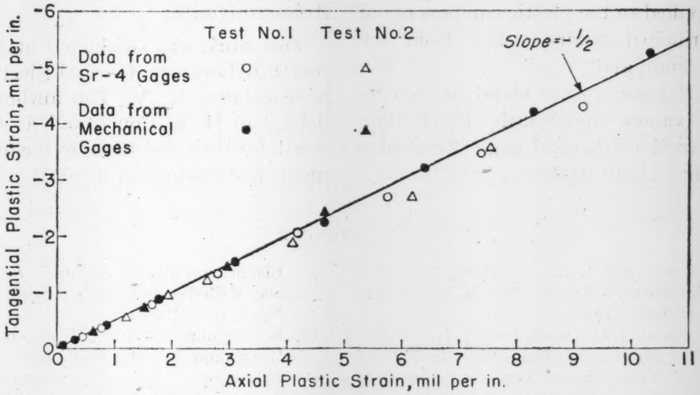

FIG. 15.—Plastic Deformation Behavior of 1-in. Round Annealed OFHC Copper Tension Bars.

liminary one because the mechanical gages involved are not of the ultra-high-precision variety, such as the interferometer or light-beam-lever types (see reference (22), for example). However, the calibration uncertainties shown in Tables I and II and in Figs. 6 and 8 indicate that the mechanical gages are accurate to perhaps a few per cent, and all the data conform to Eqs. 7 and 8 within the limits of a few per cent except for the last empty circle in Fig. 5. In other words, the second-order of precision that was obtained is probably good enough to show the validity of Eqs 7 and 8 for many practical purposes.

When the specimens were unloaded after a plastic tensile strain of only 40 × 10^{-6}, the unloading line was straight and parallel to the initial elastic line (Fig. 12), but after a plastic strain of several mils per inch or more the unloading line was quite curved and a pronounced hysteresis loop was observed upon cycling the load (Fig. 16). A similar loop in cold-stretched stainless steel has been observed (5), but this hysteresis effect was found not to be related to anelastic behavior. As discussed earlier, when such hysteresis occurs the permanent plastic strain is different from the offset plastic strain and Eqs 2 and 8 have to be handled as separate cases when considering their validity.

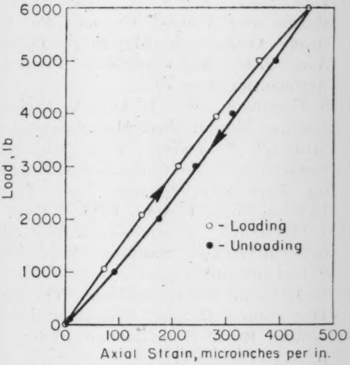

FIG. 16.—Hysteresis Curve Obtained by SR-4 Gage After 1 Per Cent Plastic Stretching. Annealed OFHC copper. 1-in. diameter.

CONCLUSIONS

1. The constant volume condition describes plastic flow behavior correctly if

it is applied to the plastic components of the principal strains (offset from the elastic line), and

2. SR-4 gages were found to indicate strain values significantly lower than those from mechanical gages for strains exceeding about 0.002.

Acknowledgment:

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AN ANALYSIS OF CERTAIN MATHEMATICAL ASSUMPTIONS UNDERLYING THE DESIGN AND OPERATION OF GAMMA-RAY SURFACE DENSITY GAGES*

By Bryant W. Pocock[1] and Walter E. Sommerman[1]

Synopsis

Literature on development of the gamma-ray surface density gage discloses conflicting reports. Investigators disagreed on best design, best type and strength of source, extent of the field of influence, effect of soil type, ease of making determinations, sensitivity of the method, and use of reference standards. A fundamental approach to the entire subject was thus undertaken. Assumptions were made on theoretical grounds only, related to known interactions of gamma radiation with matter. An equation covering gamma-ray density measurement was developed and subjected to rigorous verification. Expressions were obtained stating best thickness of lead absorber, relation between density and total linear attenuation coefficient, certain limitations on the use of standards, and effect of source strength. Magnitudes of certain functions in the equation were measured experimentally, and a predicted calibration curve was established. A gage was constructed, based on the assumptions, designed to operate most efficiently within a range of densities between 60 and 180 lb per cu ft. A nominally 5-millicurie sealed source of cesium-137 was used, separated from a TGC 2 counter tube by 6 in. of lead. Weight of the gage was 10 lb. The gage was used to measure densities of various materials which ranged in bulk density from that of air to that of lead. Similarity of the experimental and predicted calibration curves indicated validity of the assumptions underlying the fundamental equation.

During the past seven years, numerous papers have appeared describing the development of equipment for measuring densities of soils by means of interactions of gamma radiation with matter. The instruments reported have been of two types: (1) the surface gage, and (2) the depth probe.

Thought-provoking questions have been raised by the authors on such considerations as sensitivity, accuracy, ease of operation, dependency on soil type, extent of the field of influence, optimum strength and energy of radioactive source, optimum counter mechanism, and use of density standards for daily reference. Far from being settled, these questions have advanced to the status of prime controversy.

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* The investigations reported comprise one phase of the program of research adopted by the Isotopes Section of the Michigan State Highway Dept. Research Laboratory at Michigan State University, in cooperation with the University of Michigan-Phoenix Memorial Project staff in Ann Arbor. A more complete report was also submitted by the senior author as a thesis for the degree of Master of Science, Michigan State University.

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In spite of these uncertainties, a number of such gages are available commercially, especially of the surface type, and certain supply and equipment firms are working on improved models. The commercial models are expensive and have met with varying degrees of acceptance.

A survey of the literature discloses that many refinements will be required before it can be said that a truly adequate gamma-ray surface density gage has been constructed. One way of approaching this problem is to undertake a critical evaluation of basic assumptions which must be appraised before a satisfactory design can be achieved.

THEORETICAL DEVELOPMENT

Preliminary Considerations:

The intensity, A_x, of gamma radiation penetrating an absorber varies closely in accordance with the relation,

$$A_x = A_0e^{-\mu x} \quad \ldots\ldots\ldots\ldots (1)$$

where:

A_0 is the intensity at zero thickness of absorber,

μ is the total attenuation coefficient of absorber, and

x is the thickness of absorber.

The precision with which a Geiger-Müller tube can measure the term A in Eq 1 will depend upon the constants or parameters associated with the experiment in question.

Conventional gamma-ray surface density gages make use of a lead shield, which separates the source from the counter tube. This assembly is placed in contact with the material, such as soil, to be measured. Total radiation received by the counter tube is that which is reflected by and transmitted through both the lead and the substrate. The gage design should be capable of efficiently distinguishing minute differences in substrate density, yet with minimum weight. Usually the density range of interest is not large.

In general, the activity detected through the substrate only, for a given thickness x of lead between source and counter tube, is a function of the density of the substrate and of the strength and energy of the source. This activity may then be given closely by the relation,

$$A_s = g(D, E)A^{f_1(D,E)}e^{f_2(D,E)x} \quad \ldots (2)$$

where E is source energy.

For a given source energy, Eq 2 becomes

$$A_s = g(D)A^{f_1(D)}e^{f_2(D)x} \quad \ldots\ldots (3)$$

where:

A_s is the activity through the substrate only, of density D, at thickness of lead absorber, x,

$g(D)$ is the coefficient of reflection of the substrate, always positive,

$f_1(D)$ is the log scattering ratio of the substrate, always positive,

$f_2(D)$ is the negative total attenuation coefficient of the substrate, and

A is the activity through the lead only at zero thickness of lead (a function of source strength).

The total intensity, A_t, received by the counter tube then will be the sum of that through the substrate and that through the lead, or,

$$A_t = g(D)A^{f_1(D)}e^{f_2(D)x} + Ae^{-\mu x} \quad ..(4)$$

It is desirable to compare the activities scattered by and transmitted through substrates of two different densities, $D = D_1$ and $D = D_2$, where $D_1 < D_2$. Experimental evidence shows that when $D_1 < D_2$, $A_{s1} > A_{s2}$ within the limits of this discussion. Exceptions to the general statement will appear subsequently.

For $D = D_1$,

$$A_{s1} = g_1 A^{f_{11}} e^{f_{21}x} \quad \ldots\ldots\ldots\ldots (5)$$

where:
$g_1 = g(D_1, E)$,
$f_{11} = f_1(D_1, E)$, and
$f_{21} = f_2(D_1, E)$.

For $D = D_2$,

$$A_{s2} = g_2 A^{f_{12}} e^{f_{22}x} \quad \ldots\ldots\ldots\ldots (6)$$

where:
$g_2 = g(D_2, E)$,
$f_{12} = f_1(D_2, E)$, and
$f_{22} = f_2(D_2, E)$.

Attainment of Maximum Efficiency:

In order that a surface density gage operate with maximum efficiency within the range of densities between D_1 and D_2, inclusive, it is necessary to maximize the extent of ΔA_s within this density range. A suitable parameter for this purpose consists of the independent variable, x.

$$\Delta A_s = A_{s1} - A_{s2} = g_1 A^{f_{11}} e^{f_{21}x} - g_2 A^{f_{12}} e^{f_{22}x}$$

Differentiating, and solving for x

$$x = \frac{\ln\left[(f_{22}g_2/f_{21}g_1)A^{f_{12}-f_{11}}\right]}{f_{21} - f_{22}} \quad \ldots\ldots (7)$$

Equation 7 gives the value x of that thickness of lead which will provide maximum ΔA_s within any given range of substrate densities. It is a most useful relation. It will be referred to henceforth as x_{opt}.

Relationship of the Substrate Attenuation Coefficients:

It is also instructive to develop the relation between the activities, A_{s1} and A_{s2}, and the total linear attenuation coefficients of the two substrate materials concerned at the value of x, $x = x_{\text{opt}}$.

$$\frac{A_{s1}}{A_{s2}} = \frac{a}{b} u^{1-c}$$

At

$$u = \left(\frac{a}{bc}\right)^{1/(c-1)}$$

$$\frac{A_{s1}}{A_{s2}} = \frac{a}{b}\left(\frac{a}{bc}\right)^{-1} = c = \frac{f_{22}}{f_{21}} \quad \ldots\ldots (8)$$

Equation 8 states that when the thickness, x, of absorber separating the source from the counter is so chosen that the difference between the activity through a substrate only, of density $D = D_1$, and that through a substrate only, of density $D = D_2$, is at a maximum ($x = x_{\text{opt}}$), the ratio of the activities through the substrates is equal to the reciprocal of the ratio of the total linear attenuation coefficients of the two substrate materials. It is significant that this relation holds true only for $x = x_{\text{opt}}$.

It can be seen from Eq 8 that the ratio of activities through any two substrates of different densities is a constant with respect to source strength at $x = x_{\text{opt}}$, although the value of x_{opt} will vary according to Eq 7. Equation 7 contains an A term, thus causing x_{opt} to be partially dependent upon source strength.

Use of the Count Rate Meter.—It is of interest that such a ratio of two different activities through substrate materials of different densities can be measured most efficiently with a count rate meter possessing a linear scale only when the latter is adjusted to read the greater activity at the upper limit of its scale. Comparisons between activities at other scale settings diminish in precision as the highest reading falls below the upper scale limit. These considerations, in addition to generally low order of precision of rate meters and difficulty of applying statistical treatments to the data derived, quite definitely indicate the superiority of scaling equipment when small differences in density must be determined.

The "Count-in-Soil to Count-in-Standard" Ratio:

The U. S. Army Corps of Engineers, in its Field Tests of Nuclear Instruments for the Measurement of Soil and Density (6),[2] reports that a "count-in-soil to count-in-standard ratio is used to eliminate variations in count that may be caused by changes in . . . source strength." The Cornell group (1) and (2) also reported results based on the ratio of activity through an unknown substrate to activity through a standard substrate. Both groups used cobalt-60 as a source of gamma radiation.

It is important to examine the validity of the use of a "count-in-soil to count-in-standard" ratio. Changes in source strength are due to the effect of half-life only, although changes in observed count rate may be a result also of background changes. If the effects of all these changes could be eliminated by the use of a standard, as suggested, this might well avoid the necessity for making periodic calibrations of equipment.

Equations 5 and 6 may be employed to yield activities through an unknown substrate, A_{s1}, and through a standard substrate, A_{s2}. Use of the ratio $\frac{A_{s1}}{A_{s2}}$ leads to Eq 8, as shown, provided $x = x_{\text{opt}}$. At other values of the term x, use of the ratio results in the following equation:

$$\frac{A_{s1}}{A_{s2}} = \frac{g_1}{g_2} A^{(f_{11}-f_{12})} e^{(f_{21}-f_{22})x} \quad \ldots . (9)$$

Since the value of A will change with time as a function of the half-life of the source used,

$$A_{t2} = A_{t1} e^{-\lambda t} \quad \ldots\ldots\ldots\ldots (10$$

where λ is the decay constant of the source.

Upon substitution in Eq 9 of the ex-

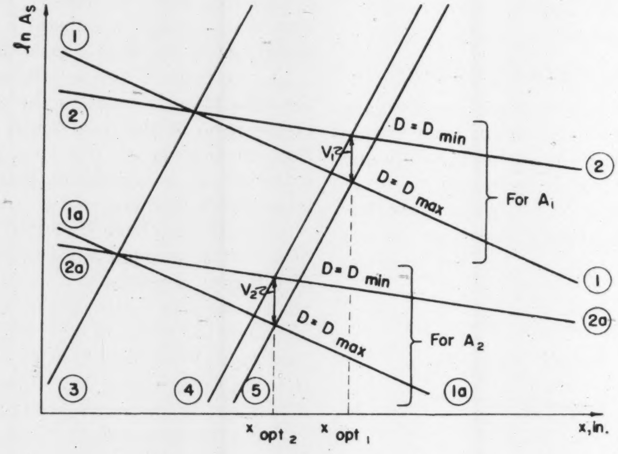

FIG. 1.—Effect of Lead Thickness on Activity Through the Substrate.

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[2] The boldface numbers in parentheses refer to the list of references appended to this paper, see p. 1357.

pression for A_{t2} obtained from Eq 10, the ratio becomes,

$$\frac{A_{s1}}{A_{s2}}$$

$$= \frac{g_1}{g_2} A_{t1}{}^{(f_{11}-f_{12})} e^{[(f_{11}-f_{12})(-\lambda t)+(f_{21}-f_{22})x]} \quad ..(11)$$

Although the value of the term t in Eq 11 is without effect where $D_{s1} = D_{s2}$ (because $A_{s1} = A_{s2}$ under these conditions), t has a significant effect where $D_{s1} \neq D_{s2}$. This leads to the conclusion that, whereas use of the ratio $\frac{A_{s1}}{A_{s2}}$ for the purpose of lessening the effect on the calibration curve of reduction of source strength with time may be justifiable on theoretical grounds, its use will not eliminate the necessity for recalibrations but merely delay this.

Effect of Lead Thickness:

From the above equations, it is possible to plot ln A_s *versus* x, as in Fig. 1, which shows activity through the substrate only as a function of the thickness of absorber separating the source from the counter tube. Curves 1 and 1(a) are for the minimum substrate density within the range of interest, and curves 2 and 2(a) are for the maximum substrate density within that range. Curves 1 and 2 are for a source (1) producing an activity A_1, and curves 1(a) and 2(a) are for a source (2) producing an activity A_2.

The antilog length V_1 gives the greatest absolute difference between curves 1 and 2, hence maximum ΔA_s for these curves, and its x-ordinate states the optimum absorber thickness for the given range of densities using source (1). Similarly, the antilog length of V_2 gives the greatest absolute difference between curves 1(a) and 2(a), hence maximum ΔA_s for these curves, and its x-ordinate states the optimum absorber thickness for the same range of densities using source (2). Sources (1) and (2) vary in strength only.

Curve 3 has the property that $A_{s1} = A_{s2}$. All curves of the type 1, 1(a), etc., intersect with corresponding curves of the type 2, 2(a), etc., along curve 3. Curve 4 contains all values of A_{s1} for which A is maximum and D is minimum within the given range of substrate densities, x being equal to x_{opt}. Curve 5 contains all values of A_{s2} for which A is minimum and D is maximum within the given range of substrate densities, x being equal to x_{opt}. The equations for curves 1 and 2 are Eqs 5 and 6, respectively, which are also valid for curves 1(a) and 2(a), etc.

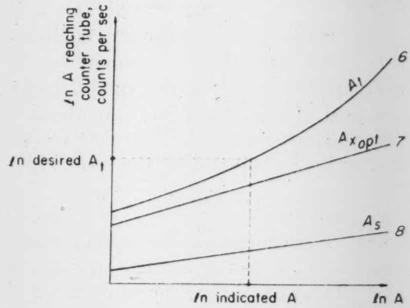

FIG. 2.—Relation Between Total Count Rate and Rate Through Lead at Zero Thickness of Lead.

Since in the case of curve 3, $A_{s1} = A_{s2}$, the equation for curve 3 is either

$$A_{s1} = g_1 \left(\frac{g_2}{g_1}\right)^{(f_{11}/(f_{11}-f_{12}))}$$

$$e \exp\left\{x\left[f_{21} + f_{11}\left(\frac{f_{22}-f_{21}}{f_{11}-f_{12}}\right)\right]\right\} \quad ..(12)$$

or

$$A_{s2} = g_2 \left(\frac{g_2}{g_1}\right)^{(f_{12}/(f_{11}-f_{12}))}$$

$$e \exp\left\{x\left[f_{22} + f_{12}\left(\frac{f_{22}-f_{21}}{f_{11}-f_{12}}\right)\right]\right\} \quad ..(13)$$

The equation for curve 4 is

$$A_{s1} = g_1 \left(\frac{f_{22}g_2}{f_{21}g_1}\right)^{(f_{11}/(f_{11}-f_{12}))} e \exp\left\{\left[f_{21} + f_{11}\left(\frac{f_{22}-f_{21}}{f_{11}-f_{12}}\right)\right] x_{\text{opt}}\right\} \quad ..(14)$$

and that for curve 5 is

$$A_{s2} = g_2 \left(\frac{f_{22}g_2}{f_{21}g_1}\right)^{(f_{12}/(f_{11}-f_{12}))} e \exp\left\{\left[f_{22} + f_{12}\left(\frac{f_{22}-f_{21}}{f_{11}-f_{12}}\right)\right] x_{\text{opt}}\right\} \quad ..(15)$$

Determination of Source Strength:

To obtain the maximum counting rate with available equipment, it is necessary to consider the rate through the lead absorber and the rate through the substrate material. The rate through the lead is established by the term,

$$Ae^{-\mu x}$$

where μ is the total attenuation coefficient of lead and x is the thickness of lead. The rate through a substrate material of density $D = D_{\min}$ in the range of interest (so that A_s is maximum within this range) is given by the term,

$$g_1 A^{f_{11}} e^{f_{21} x}$$

in which x is that thickness of lead absorber, $x = x_{\text{opt}}$, which will provide maximum ΔA_s within the density range. The total count rate, A_t, is given by the relation,

$$A_t = \underbrace{\left(\frac{f_{22}g_2}{f_{21}g_1}\right)^{(-\mu/(f_{21}-f_{22}))} A^{[1-(\mu(f_{12}-f_{11}))/(f_{21}-f_{22})]}}_{\substack{\| \\ A_x}} + \underbrace{g_1\left(\frac{f_{22}g_2}{f_{21}g_1}\right)^{(f_{21}/(f_{21}-f_{22}))} A^{[f_{11}+f_{21}(f_{12}-f_{11})/(f_{21}-f_{22})]}}_{\substack{\| \\ A_{sx}}} \quad ..(16)$$

$$A_t = \qquad A_x \qquad A_{sx}$$

where $x = x_{\text{opt}}$.

It is seen that Eq 16 is the equation of a curve 6, which is the sum of two separate curves, 7 and 8. These curves can be plotted on logarithmic coordinate paper, as shown in Fig. 2. In the case of curves 7 and 8, the coefficient gives the intersection with the ln A_t-axis at $A = 1$, and the exponent gives the slope.

Once a value of A_t has been selected as convenient for the equipment to be used, the value of A for this value of A_t at $x = x_{\text{opt}}$ can be read from the graph. To determine the indicated source strength to be used, this value of A should be divided by the efficiency of the detection unit in the geometry under consideration. It should be emphasized that the value of μ in Eq 16 has been modified by varying the source-counter distance, and must be determined by experiment.

TABLE I.—SUBSTRATE MATERIALS USED IN EXPERIMENT 1.

Material	Bulk Density, lb per cu ft
Concrete	150.2
Sand	93.8
Clay	63.09
Wood	29.2

EXPERIMENTAL DEVELOPMENT

Equipment and Procedure:

A series of laboratory experiments was designed to evaluate the optimum thickness of lead absorber, x_{opt}; the magnitude of the functions, f_{11}, f_{21}, f_{12}, and f_{22}; and the amount of the activity A. A 5-millicurie sealed cesium-137 source was purchased, to be used in conjunction with a TGC 2 GM counter tube. It was decided to evaluate x_{opt} within the range of substrate densities between 60 and 180 lb per cu ft, inclusive.

A number of 3 by 9 by $\frac{1}{8}$-in. lead plates were prepared for use as separators be-

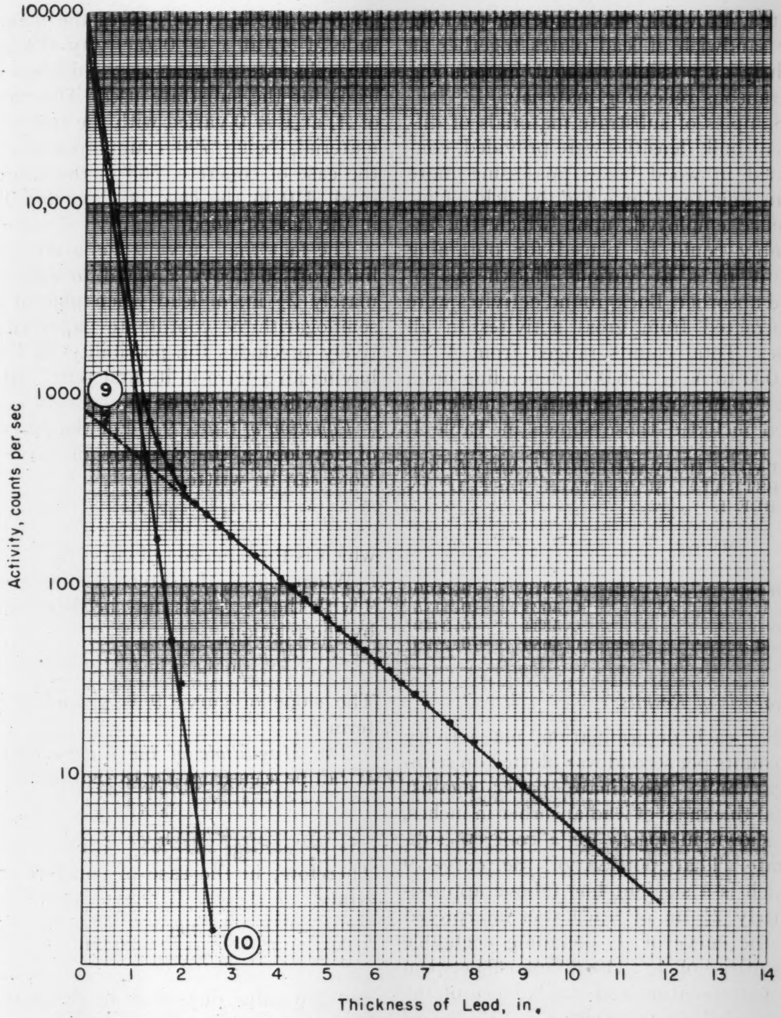

FIG. 3.—Laboratory Count Rate on Concrete Substrate *versus* Thickness of Lead.

tween the source and the counter tube. The assembly was mounted in such a manner that the number of lead plates, standing upright on their long edges between the source and the counter, could be varied.

Rather than keep the source-to-counter distance constant for all numbers of lead absorber plates, this distance was allowed to change with the total lead thickness. In this way, for each thickness of lead, the typical geometry of a gamma-ray surface density gage containing that thickness of lead absorber was approximated. Provision was made for packing the sandwich of lead plates together as tightly as possible without introducing extraneous reflecting materials.

Samples of substrate materials of different bulk densities were provided, contained in wood boxes possessing more than infinite volume for the field of influence employed, upon which the assembly could be placed for obtaining count rates at various thicknesses of lead absorber. Background activites were subtracted from gross activites in all cases. Total counts ranged from $N = 16{,}000$ to $N = 256{,}000$, depending upon the count rate. Substrate materials used included those shown in Table I.

TABLE II.—VALUES OF f_1 AND f_2 FOR SUBSTRATE MATERIALS LISTED IN TABLE I.

Material	f_1	f_2
Concrete	0.5916	−0.5040
Sand	0.5623	−0.4115
Clay	0.5334	−0.3442
Wood	0.4689	−0.2290

Analysis of Results:

For each determination, net activity in counts per second was plotted on semilogarithmic coordinate paper against total thickness of lead absorber in inches, as shown in Fig. 3 for a concrete substrate. That portion of the curve 9 which is a straight line (above approximately $2\frac{1}{4}$ in. of lead) was interpreted as the curve of A_s including negligible quantities of A_x. The difference between its extrapolation and the balance of the curve below approximately $2\frac{1}{4}$ in. of lead (curve 10) was interpreted to be the curve of A_x only.

The intersection of curve 10 with the ln A-axis gives the magnitude of A for the substrate used. This value was found to be 79,000 counts per sec in the case of all substrate materials listed in Table I.

The intersection of curve 9 extrapolated to the ln A-axis gives the magnitude of A_s at $x = 0$ (activity through the substrate only at zero thickness of lead) for the substrate used. The value of A_s at $x = 0$ varied with the substrate material, being 790 counts per sec in the case of concrete, 568 in the case of sand, 410 in the case of clay, and 198 in the case of wood.

The fact that curve 9 has a straight-line portion above x equal to approximately $2\frac{1}{4}$ in. of lead when plotted on semilogarithmic coordinate paper effectively precludes the possibility of Eq 2 having an x-term in its first term, either as a coefficient of or as an exponent of A.

Equation of Curve 9.—For the purpose of developing the equation of curve 9, Eq 3 can be written,

$$A_s = A^{f_1} e^{f_2 x} \quad \text{(17)}$$

which is the equation of curve 9.

When $x = 0$, the intersection of curve 9 with the ln A-axis may be determined by the relation,

$$\ln A_s = \ln A^{f_1} \quad \text{(18)}$$

The slope of curve 9 is given by the term f_2.

The Magnitude of the f Functions.—It can be seen from Eq 18 that at $x = 0$,

$$f_1 = \frac{\ln A_s}{\ln A} \quad \text{(19)}$$

Therefore, in the case of concrete substrate,

$$f_1 = \frac{\ln 790}{\ln 79{,}000} = 0.5916 \quad \text{(20)}$$

and the value of f_2 may be determined between any two points, as, for example, $x = 6$ and $x = 8.7$. Then,

$$f_2 = \frac{2.30259 - 3.66356}{2.7} = -0.5040 \quad \text{(21)}$$

Similarly, curves corresponding to curve 9 for a concrete substrate may be plotted from data derived from use of the other substrate materials listed in Table I. The values of the functions

f_1 and f_2 calculated from these data in a manner similar to that outlined in Eqs 20 and 21 are given in Table II.

A Predicted Calibration Curve.—The values of the functions f_1 and f_2 shown in Table II were plotted on logarithmic coordinate paper against the bulk densities of the substrate materials, as shown in Fig. 4. The values of f_1 and f_2 for wood fall slightly below their corresponding curves, which may be attributed to the probability that the volume of the wood sample used was insufficiently infinite for the geometry employed.

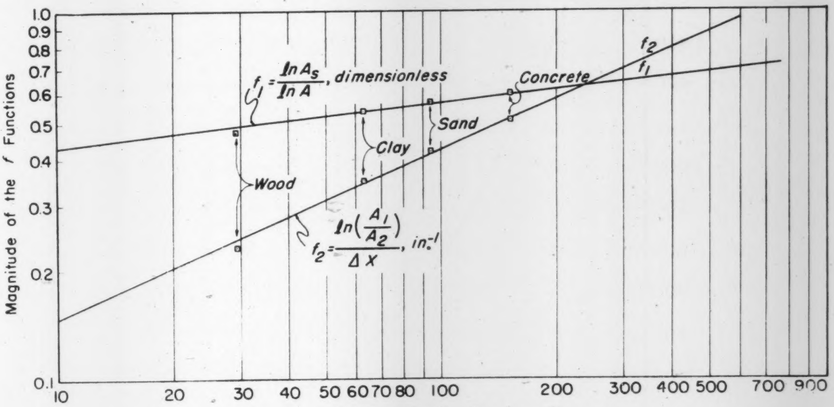

FIG. 4.—*f* Functions *versus* Substrate Density.

The curves for f_1 and f_2 in Fig. 4 were used to obtain values for these functions at other densities to establish a quasi-theoretical calibration curve for an instrument designed on the basis of the above data. This predicted calibration curve is shown in Fig. 5.

DEVELOPMENT OF A GAMMA-RAY SURFACE DENSITY GAGE BASED ON THE FOREGOING DATA

Construction of the Gage:

After substitution of the values of f_{11} and f_{21} at $D = 60$ lb per cu ft and of the values of f_{12} and f_{22} at $D = 180$ lb per cu ft, taken from the f_1 and f_2 curves of Fig. 4, it was found from Eq 7 that the magnitude of x_{opt} was 6.348 in. using the 5-millicurie source of cesium-137. However, an accurate plot of Fig. 1 showed that the magnitude of ΔA_s at $x = 6.000$ in. was 17.0 counts per sec, compared with a magnitude of 17.1 counts per sec at $x = 6.348$ in. This difference in magnitude of ΔA_s, therefore, was only 0.58 per cent of its magnitude at $x = x_{opt}$. By accepting a value of 6.000 in. for x_{opt}, it was found that a saving of 5.482 per cent could be realized in the weight of the lead absorber.

Accordingly, a gage was designed on the basis that $x_{opt} = 6.0$ in., as illustrated in Fig. 6 using the 5-millicurie sealed cesium-137 source of gamma radiation. Housing for the assembly was constructed of 28-gage monel metal arranged in the shape of a box with a sliding, removable cover. Total weight of the gage was 10 lb.

Laboratory and Field Applications:

Count rates obtained with the instrument were plotted against bulk densities for a number of materials. Substrates employed included air, wood shavings, wood boards, paper, concrete, steel, and lead, and various soils under several degrees of compaction. The soils included laboratory and field samples. In the laboratory, they were contained in wood boxes measuring 20 by 24 in. These were filled to a depth of approximately 6 in. and struck off flush with the tops. Control densities were determined by volume and weight measurements on a wet basis.

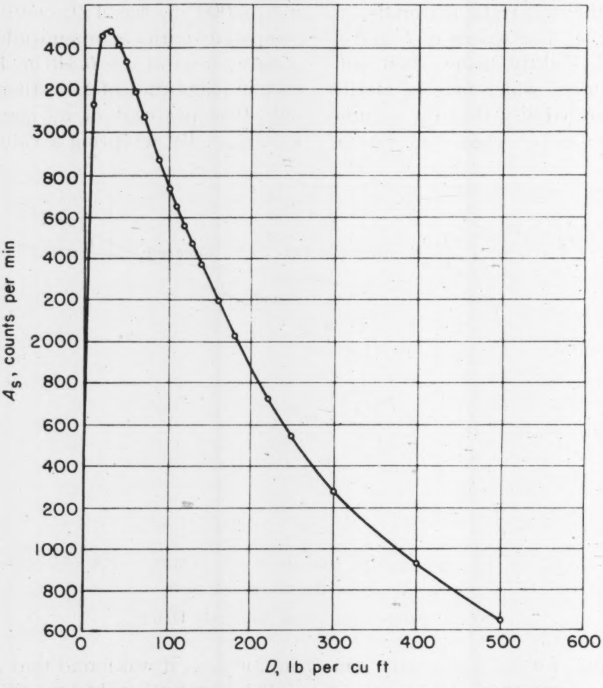

FIG. 5.—Predicted Calibration Curve Based on $A_s = gA^{f_1(D)}e^{f_2(D)x}$ $= (79{,}000^{f_1(D)}e^{6f_2(D)})60$

Field gage measurements were made with the soil *in situ*. After gage measurements had been completed, samples were taken at the same sites for conventional density determinations and for laboratory analyses. Occasionally, the soil was tamped, and both types of density determination were repeated to evaluate the effect of tamping on soil compaction. It was found that tamping increased the density of soil No. 21 by approximately 16 lb per cu ft.

Conventional density determinations made at the site were based on volume-weight relationships taken immediately, by use of a portable balance and the so-called "can" technique. Both tops and bottoms were removed from 12-oz No. 1 cans, and one end of each can was ground sharp. It was found possible to insert the can into the soil with minimum

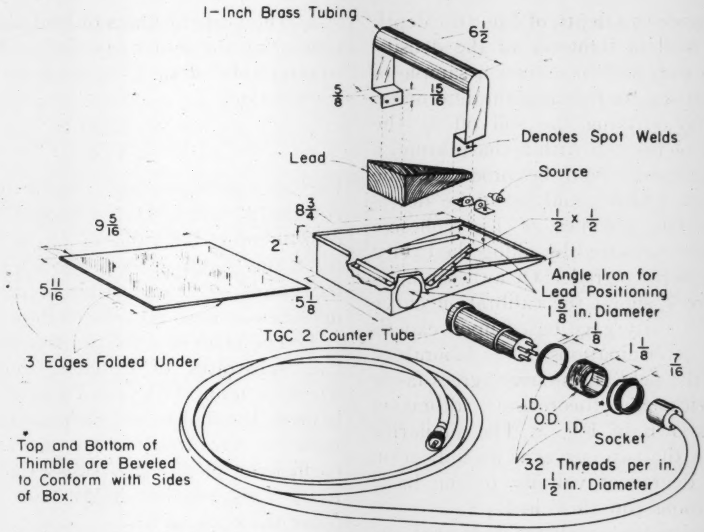
1-Inch Brass Tubing
6 1/2
5/8
1
15/16
Denotes Spot Welds
Lead
Source
8 3/4
9 5/16
2
1/2 x 1/2
1
5 11/16
5 1/8
Angle Iron for
Lead Positioning
1 5/8 in. Diameter
TGC 2 Counter Tube
1/8
1 1/8
7/16
3 Edges Folded Under
I.D.
O.D.
I.D.
Socket
32 Threads per in.
1 1/2 in. Diameter
* Top and Bottom of
Thimble are Beveled
to Conform with Sides
of Box.

FIG. 6.—Surface Density Gage Constructed by Michigan State Highway Department Isotopes Section.

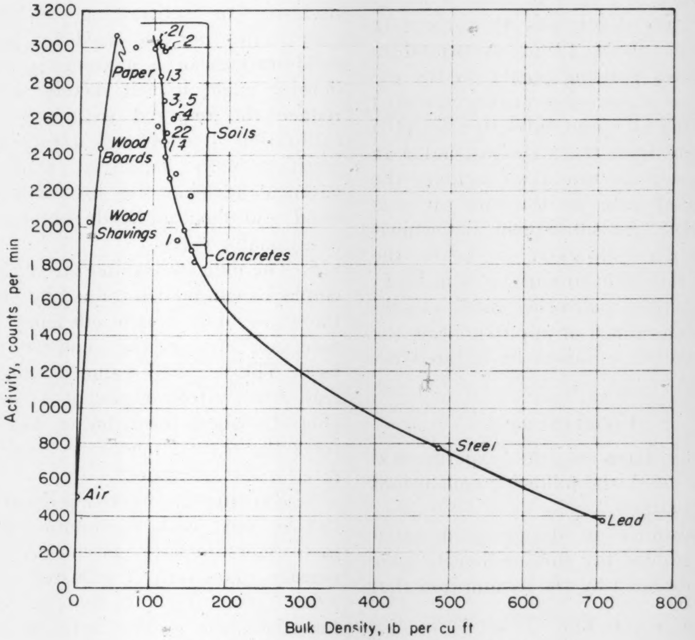

FIG. 7.—Experimental Calibration Curve.

disturbance to a depth of 4 in. (the depth of the field of influence of the density gage in soil) and to extract a sample of sufficient size for independent determinations. By striking the soil off at the bottom of the can with a sharp knife, a right cylinder could be produced, the height of which could easily be determined. The volumes of less cohesive soils were measured by filling the cavity with uniformly graded Ottawa sand.

Figure 7 shows the calibration curve for the density gage based upon experimental determinations in the laboratory and in the field. This curve agrees fairly well with the theoretical calibration curve shown in Fig. 5. The similarity between the two curves is an indication of the degree of validity of the basic assumptions contained in Eq 2.

Experimental evidence indicated, as noted above, that the depth of the field of influence in soils was about 4 in. In concrete, the depth was 3½ in., and in air it was about 12 in. A procedure adopted for plotting points on the experimental calibration curve consisted of referring all count rates to a selected count rate with the gage suspended in air. The selected rate in air included the background rate at the time it was determined. As subsequent determinations in air fell above or below the selected rate, due to variations in background activity, all other rates on substrates determined at approximately the same time were raised or lowered accordingly.

CONCLUSIONS

On the basis of the experimental evidence cited, the following conclusions appear justified:

1. Assistance in designing a satisfactory gamma-ray surface density gage can be obtained by the assumption that

$$A_s = g(D, E)A^{f_1(D,E)}e^{f_2(D,E)x} \quad \ldots . (2)$$

2. The best thickness of lead absorber separating the source and Geiger-Müller counter tube of such a gage is given by the relation,

$$x_{\text{opt}} = \frac{\ln\,[(f_{22}g_2/f_{21}g_1)A^{f_{12}-f_{11}}]}{f_{21} - f_{22}} \quad \ldots\ldots (7)$$

For a gage employing 5 millicuries of cesium-137 and a TGC 2 Geiger-Müller counter tube, the value of x_{opt} is 6.4 in. when the gage is intended to operate at maximum efficiency within a range of densities between 60 and 180 lb per cu ft.

3. The ratio of activities through any two substrates of different densities, when optimum thickness of lead absorber is used, is equal to the reciprocal of the ratio of the total linear attenuation coefficients of the two substrate materials. This relation holds true at no other thickness of lead.

4. Use of a "count-in-soil to count-in-standard" ratio will not eliminate the necessity of subtracting background count rates nor the need for periodic recalibrations of a gamma-ray surface density gage; it will merely serve to reduce the required frequency of recalibration. Use of such a ratio is not justified with long-lived sources of gamma radiation. Its use with short-lived and medium-lived isotopes is of questionable value.

5. The indicated source strength to be employed can be determined by dividing the value of A by the efficiency of the detection unit in the geometry to be used. The required value of A can be read directly from a plot of A_t *versus* A (Fig. 2), where the value of A_{tot} represents the desired maximum total count rate for the instrument.

6. Rate meters employing linear scales can be used with maximum efficiency in conjunction with gamma-ray surface density gages only if adjusted so that, when comparing the densities of two materials, the greater activity is read

at the upper scale limit. The superiority of scaling equipment over count rate meters for use with density gages appears to be indicated.

7. The need for a commercially available, portable, battery-operated scaler of modest cost and of rugged, though light, design is definitely indicated.

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(13) Gerhart Friedlander and Joseph W. Kennedy, "Introduction to Radiochemistry," John Wiley & Sons, Inc., New York, N. Y., p. 170 (1949).

(14) John L. Putnam, "Industrial Uses of Radioisotopes," Geneva Conference, Aug., 1955.

SUMMARY OF PROCEEDINGS OF THE SYMPOSIUM ON RADIATION EFFECTS ON MATERIALS, VOL. 3

This Symposium on Radiation Effects on Materials is the third and last of a series which has been sponsored jointly by ASTM and the Atomic Industrial Forum. The papers presented only new information in the field, in contrast to the first and second symposiums which included both declassified and new data. Major items of current interest were presented, divided into the following categories: (1) dosimetry techniques; (2) irradiation facilities and techniques; and (3) studies of irradiation effects.

The papers in this symposium were presented at the eleventh and thirteenth sessions, June 24, 1958, of the Sixty-first Annual Meeting of the Society, Boston, Mass. The symposium was developed under the joint auspices of the Subcommittee on Radiation Effects of the Atomic Industrial Forum and ASTM Committee E-10 on Radioisotopes and Radiation Effects.

Messrs. J. H. Kittel, Argonne National Laboratory, Lemont, Ill., and A. N. Tschaeche, Reynolds Electric Co., Las Vegas, Nev., acted as Symposium Co-Chairmen, and both presided over the morning session. The afternoon session was presided over by Mr. Kittel and Mr. C. R. Sutton, The International Nickel Co., Inc., New York, N. Y.

The papers presented were:

Introduction—A. N. Tschaeche and J. H. Kittel

Dosimetry Considerations

Dosimetry Techniques for Gamma and Reactor Radiation Fields—Paul Schall and J. F. Kircher

Irradiation Facilities and Techniques

Irradiation Facilities in NRU—G. C. Laurence

The Engineering Test Reactor as an Irradiation Facility—R. L. Doan

An Integrated Facility for Study of Effects of Nuclear Radiation on Materials—John L. Colp and A. William Snyder

An In-Pile Fatigue Testing Apparatus—E. E. Drucker

Studies on Radiation Effects

Reactor Pressure Vessel Design for Nuclear Applications—N. Balai, T. L. Kettles, and R. E. Bailey.

Effects of Irradiation on the Type 347 Stainless Steel Flow Separator in the EBR-I Core—R. E. Bailey and M. A. Silliman

Radiation Damage Effects on Reactor Control Materials—W. E. Ray, W. K. Anderson, and D. N. Dunning

Radiation Behavior of Fuel Materials for Sodium Graphite Reactors—B. R. Hayward, L. E. Wilkinson, and C. C. Woolsey

Irradiation of Uranium-Fissium Alloys and Related Compositions—Karl F. Smith

Effects of Nuclear Radiation on Natural Quartz Piezoelectric Crystals: Preliminary Investigation—F. E. Graham and A. F. Donovan

Effects of High Neutron and Gamma Fluxes on Transmission Characteristics of Some Optical Glasses—John L. Colp and H. N. Woodall

These papers with discussion have been issued as *ASTM Special Technical Publication No. 233* entitled "Symposium on Radiation Effects on Materials—Volume 3."

SUMMARY OF PROCEEDINGS OF THE SYMPOSIUM ON MATERIALS RESEARCH FRONTIERS

Tailoring materials to fit specific needs was the keynote of the Symposium on Materials Research Frontiers. Molecular engineering and physical metallurgy were among the means to this end discussed in the papers presented.

The papers in this symposium were presented at the twenty-first and twenty-fourth sessions of the Sixty-first Annual Meeting of the Society in Boston, Mass., June 25, 1958. The symposium was developed under the joint auspices of the ASTM Administrative Committee on Research and the New England District Council Committee.

Mr. Walter J. Smith, of Arthur D. Little, Inc., and Mr. Miles N. Clair, of The Thompson & Lichtner Co., Inc., acted as Symposium Chairmen. Mr. R. T. Kropf, Belding Heminway Co., Inc., and Mr. Walter J. Smith presided over the morning session. Mr. K. B. Woods, School of Civil Engineering, Purdue University, and Mr. A. G. H. Dietz, Massachusetts Institute of Technology, presided over the afternoon session.

The papers presented were:

Tailoring the Properties of Materials—E. P. Stevenson
Molecular Engineering—Arthur von Hippel
Instruments in Materials Research—Bruce H. Billings
Materials in the Nuclear Age—John J. Antal
Recent Advances in Polymer Research—Herman Mark
Modern Liquid Fuels—Arthur L. Lyman
New Advances in Physical Metallurgy—W. R. Hibbard
Recent Developments in Glass Research—W. W. Shaver

The papers in this symposium, with the exception of the papers by Herman Mark and Bruce H. Billings have been issued as *ASTM Special Technical Publication No. 243* entitled "Symposium on Materials Research Frontiers." An abstract only of the paper by Arthur von Hippel is included in the symposium volume.

SUMMARY OF PROCEEDINGS OF THE SYMPOSIUM ON RADIOACTIVITY IN INDUSTRIAL WATER AND INDUSTRIAL WASTE WATER

In this Symposium on Radioactivity in Industrial Water and Industrial Waste Water problems in the reactor plant proper are discussed, the associated waste water processing and control methods described, and methods of analysis for radionuclides in industrial water and industrial waste water presented, including radiation hazards. The effect of nuclear operations on the environment and analysis of samples pertaining thereto are included.

The papers in this symposium were presented June 26, 1958, at the thirty-eighth session of the Sixty-first Annual Meeting of the Society, Boston, Mass. The symposium was sponsored by ASTM Committee D-19 on Industrial Water. Mr. A. R. Belyea, Consolidated Edison Co. of New York, Inc., acted as symposium chairman and presided over the session.

The papers presented were:

Introduction—A. R. Belyea
Radioactivity and Purity Control of APPR Primary Water—A. Louis Medin
Radioactive Waste Processing Control, Shippingport Atomic Power Station—S. F. Whirl and J. A. Tash
Test Methods for Radioactivity Hazards in Industrial Waters—C. J. Munter
Analysis for Radionuclides in Aqueous Wastes from an "Atomic" Plant—B. Kahn, D. W. Moeller, T. H. Handley, and S. A. Reynolds
Analysis of Environmental Samples for Radionuclides—D. L. Reid
Summation—J. M. Seamon

These papers with discussion have been issued as *ASTM Special Technical Publication No. 235* entitled "Symposium on Radioactivity in Industrial Water and Industrial Waste Water." The published volume includes a paper not presented at Boston: "Analysis of Radioactivity in Surface Waters—Practical Laboratory Methods," by L. R. Setter, G. R. Hagee, and C. P. Straub, which was first published in the ASTM BULLETIN, No. 227, Jan., 1958, p. 35 (TP 1).

SUMMARY OF PROCEEDINGS OF THE SYMPOSIUM ON SOME APPROACHES TO DURABILITY IN STRUCTURES

With the advent of curtain wall construction the subject of durability in structures has become more important than before when resistance to weathering of the exposed sections of buildings was considered a major item in construction and maintenance. The fact that there is no single approach to achieving durability is ably brought out in the papers presented in this Symposium. This Symposium was sponsored by Subcommittee II on Durability of ASTM Committee E-6 on Methods of Testing Building Constructions.

The papers and discussions in this Symposium were presented at the Third Session of the Sixty-first Annual Meeting of the Society, held in Boston, Mass., June 23, 1958, Mr. E. C. Shuman, Owens-Corning Fiberglas Corporation, Toledo, Ohio, chairman of the Symposium Committee presiding over the session.

The papers included in the Symposium are:

Introduction—E. C. Shuman
Some Factors Affecting Durability of Structural Clay Product Masonry—P. V. Johnson and H. C. Plummer
Laboratory Testing and the Durability of Concrete—T. B. Kennedy
Durability Tests of Structural Sandwich—E. W. Kuenzi and L. W. Wood
The Durability of Buildings—R. F. Legget and N. B. Hutcheon
Effect of the Atmosphere on Masonry and Related Materials—J. W. McBurney
Relation Between Actual and Artificial Weathering of Organic Material—F. W. Reinhart
Closing Comments—E. C. Shuman

These papers with discussion were issued as *ASTM Special Technical Publication No. 236* entitled "Symposium on Some Approaches to Durability in Structures."

SUMMARY OF PROCEEDINGS OF THE SYMPOSIUM ON BULK SAMPLING

This Symposium was sponsored by ASTM Committee E-11 on Quality Control of Materials. Its purpose is to provide a further understanding of sampling for those in charge and responsible for preparing procedures for sampling.

The papers and discussion in this Symposium were presented at the Fifth Session of the Sixty-first Annual Meeting of the Society, held in Boston. Mass., June 23, 1958. Mr. B. H. Lloyd, Price Waterhouse and Co., acted as Symposium Chairman and presided over the evening session, and Mr. Simon Collier, Johns-Manville Corp., presided over the afternoon session.

The papers included in the Symposium are:

Measurement Error Considerations in Bulk Sampling with Special Reference to the Sampling of Fertilizer—Acheson J. Duncan

An Interpretation of the Enos Coal Sampling Data as an Example of Bulk Sampling—B. A. Landry

The Effect of Increment Weight on Sampling Accuracy—W. M. Bertholf

The Purposes of Sampling—W. E. Deming

Studies in Ore Car and Abrasive Grain Sampling Variation—R. S. Bingham, Jr., J. L. Gioele, and V. B. Shelburne

These papers with discussion, with the exception of the paper by W. E. Deming, were issued as *ASTM Special Technical Publication No. 242*, entitled "Symposium on Bulk Sampling."

SUMMARY OF PROCEEDINGS OF THE SYMPOSIUM ON PAPER AND PAPER PRODUCTS

NEW DEVELOPMENTS WITH ACCOMPANYING REQUIREMENTS FOR NEW TESTING METHODS

Committee D-6 on Paper and Paper Products recently recognized that the well-established test methods of the paper industry would need to be critically reviewed in order to keep pace with the increased use of synthetic resin binders and fibers, new surface finishes, and new methods of paper manufacture. As a result the committee decided to sponsor a broad scope symposium on new developments in the industry in order to stimulate greater interest in the problems of revising the existing standard methods for paper testing.

The symposium was presented at the thirty-fourth and thirty-sixth sessions of the Sixty-first Annual Meeting of the Society held on June 26, 1958, in Boston, Mass. H. A. Birdsall, Bell Telephone Laboratories, Inc., acted as Symposium chairman and he and F. F. Newkirk, American Sisalcraft Corp., presided over the two sessions.

The papers included in the Symposium are:

Introduction—H. A. Birdsall
Some Historical Developments in Paper Testing—W. R. Willets and F. R. Marchetti
Testing of Synthetic Fiber Papers—F. H. Koontz and J. K. Owens
Nonwoven Fabrics and Synthetic Fiber Papers: Technology and End Uses—J. T. Taylor and P. J. McLaughlin
New Developments in the Internal Bonding of Paper—K. W. Britt
A New Cotton Paper Furnish—Properties, Applications, Identification—J. A. Harpham
Clupak Paper—A New Type of High Stretch Paper—Its Manufacture and Performance—R. J. Diaz
Relative Humidity Measurements in Package Materials Testing—B. H. Schrier and P. K. Wolper

These papers with discussion were issued as *ASTM Special Technical Publication No. 241*, entitled "Symposium on Paper and Paper Products—New Developments with Accompanying Requirements for New Testing Methods."

SUMMARY OF PROCEEDINGS OF THE SYMPOSIUM ON STABILITY OF DISTILLATE FUEL OILS

This Symposium on Stability of Distillate Fuel Oils sponsored by Technical Committee E on Burner Fuel Oils of ASTM Committee D-2 on Petroleum Products and Lubricants reviews the present situation with regard to stability and its effect on performance of home-heating fuel oils.

The papers in this Symposium were presented at a committee-sponsored session during the Sixty-first Annual Meeting of the Society, held in Boston, Mass., June 25, 1958. Mr. W. deB. Bertolette, E. I. du Pont de Nemours and Co., Inc., was Symposium Chairman.

All the papers presented, except G. T. Kaufman's paper, "Burning Today's Oil," are included in the publication:

Trials and Tribulations of Large Development Oil Heating—N. M. Edmands
Predictive Type Tests for Storage Stability and Compatibility of Diesel Fuels—R. T. Jones and J. W. MacDonald
Requirements for Dependable Performance of Domestic Oil Burner Nozzles—E. O. Olson
Do We Need a Stability Specification for No. 2 Heating Oil?—Robert Gray
A Review of the Distillate Fuel Stability Problem—E. A. Elmquist
How Distillate Fuel Stability Is Measured and Controlled—W. L. Clinkenbeard
Distillate Fuel Incompatibility—C. C. Ward and F. C. Schwartz
Distillate Fuel Oil Gel—F. R. Dunn, Jr. and R. W. Sauer

These papers were issued as *ASTM Special Technical Publication No. 244* entitled "Symposium on Stability of Distillate Fuel Oils."

SUMMARY OF PROCEEDINGS OF THE SYMPOSIUM ON EFFECT OF OZONE ON RUBBER

This Symposium on Effect of Ozone on Rubber was sponsored by Committee D-11 on Rubber and Rubber-Like Materials. One large user group, the automotive industry, has been vitally interested in the ozone resistance of rubber parts both before and after the automobile moves into the customer's hands. Today, we do not have the complete answer to the problem, but the papers presented in this Symposium indicate a good rate of progress during the past few years.

The papers and discussion in this Symposium were presented at a two-session meeting, February 10, 1958, during the Society's Committee Week in St. Louis, Mo., G. C. Maassen, R. T. Vanderbilt Co., Inc., Chairman of the Symposium, presiding over the sessions.

The papers included in the Symposium are:

Introduction—G. C. Maassen
Rubber and Its Environment—A. J. Haagen-Smit
A Study of the Action of Ozone with Polybutadiene Rubbers—E. R. Erickson, R. A. Berntsen, E. L. Hill, and Paul Kusy
The Reaction of Ozone with Rubber—Harold Tucker
Ozone Resistance of Elastomeric Vulcanizates—Z. T. Ossefort
Chemical Antiozonants and Factors Affecting Their Utility—William L. Cox
Prevention of Ozone Attack on Rubber by Use of Waxes—S. W. Ferris, S. S. Kurtz, Jr., and J. J. Sweely
Comparison of Accelerated and Natural Tests for Ozone Resistance of Elastomers—G. N. Vacca
Quantitative Measurement of Rate of Ozone Cracking—A. G. Veith
Report on Interlaboratory Ozone Test Program of ASTM Committee D-11, Subcommittee XV, 1957—A. G. Veith

These papers with discussion were issued as *ASTM Special Technical Publication No. 229* entitled "Symposium on Effect of Ozone on Rubber."

SUMMARY OF PROCEEDINGS OF THE SYMPOSIUM ON INSTRUMENTATION IN ATMOSPHERIC ANALYSIS

This Symposium on Instrumentation in Atmospheric Analysis was sponsored by Subcommittee III on Instrumentation of ASTM Committee D-22 on Methods of Atmospheric Sampling and Analysis. Instruments offer special advantages in atmospheric analysis because they can be constructed to operate unattended for various periods and can provide a chart of concentration of pollutant over a period of time. For this reason and others, there has been continuing effort in Committee D-22 in developing standards for proven types of instrument. This Symposium provides useful information on performance and utility of several types of instrument for air analysis.

The papers in this Symposium were presented at a special session of the Sixty-first Annual Meeting of the Society held in Boston, Mass., June 23, 1958. M. D. Thomas, Stanford Research Institute, acted as chairman presiding over the session.

The papers included in the Symposium are:

Light Scattering Instrumentation for Particle Size Distribution Measurements—Chester T. O'Konski, M. D. Briton and W. I. Higuchi
Filtration Methods for Evaluation of Aerosol Contaminants—Morris Katz and H. P. Sanderson
Relative Merits of Gas Chromatography, Colorimetry and Spectrometry for Air Pollution Measurements—Lewis H. Rogers
The SRI Fluoride Recorder—Moyer D. Thomas, Gilbert A. St. John, and Saul W. Chaikin

These papers were issued as *ASTM Special Technical Publication No. 250* entitled "Symposium on Instrumentation in Atmospheric Analysis."

SUMMARY OF PROCEEDINGS OF THE SYMPOSIUM ON CLEANING OF ELECTRONIC-DEVICE COMPONENTS AND MATERIALS

The Symposium on Electronic-Device Components and Materials was sponsored by ASTM Committee F-1 on Materials for Electron Tubes and Semiconductor Devices. Current progress in the control of contamination during assembly of electronic-device components and materials was discussed at four sessions of this two-day Symposium, held in Philadelphia, October 13–14, 1958.

Mr. F. J. Biondi, Bell Telephone Laboratories, was the Symposium Chairman; Mr. S. A. Standing, Raytheon Manufacturing Co., presided over the first two sessions, Mr. C. W. Horsting, Radio Corporation of America, acting as moderator; and Mr. S. Umbreit, Radio Corporation of America, presided over the third and fourth sessions, Mr. A. P. Haase, General Electric Co., being moderator.

The papers included in the Symposium are:

Introduction—F. J. Biondi

Physical and Organic Contaminants

Measuring and Controlling Dust—P. R. Pondy and G. E. Helmke

Planning and Operating a Clean Shop—W. T. Dyall and L. C. Herman

An Ultrasonic System for Eliminating Physical Contaminants from Electron Devices—D. E. Koontz and I. Amron

"Operation Snow White"—An Approach to Higher Reliability in Receiving Tubes—K. D. Johnson

Detection, Removal, and Control of Organic Contaminants in the Production of Electron Devices—D. O. Feder and D. E. Koontz

Cleanness Factors in Mechanical Processes and Etching of Semiconductors—J. W. Faust, Jr.

Gaseous Contaminants

Design and Use of a Mass Spectrometer to Study Gas Problems in Electron Device Development—E. J. Becker

Use of the Mass Spectrometer to Evaluate the Effectiveness of Processing Tube Materials—R. W. Griessel

Carbon as an Indicator of Gas Content in Metallic Tube Components—D. R. Kerstetter

Method of Analysis and Control of Gas Atmospheres Used in the Processing of Electronic Parts and Devices—M. J. Elkind and D. R. Benn

The Adsorption and Desorption Characteristics of Conductive Graphite Coatings—Herman H. Stern and D. Joseph Donahue

Hydrogen Absorption by Tube Parts Due to Various Processing Procedures—David Lichtman

Chemically Combined Contaminants

Cathodic Electrocleaning of Molybdenum Wire Prior to Gold Plating—Robert W. Etter

The Preparation of Ultraclean Electron-Tube Components by Chemical Etching—D. E. Koontz, C. O. Thomas, W. H. Craft, and I. Amron

Radiotracers in Evaluating Parts Cleaning—Morris N. Slater and D. Joseph Donahue

Effects of Parts Cleaning on Tube Performance—J. C. Hickle and S. C. Crawford

Thermionic Emission from Oxide Cathodes as Related to Glass—H. E. Kern and E. T. Graney

An Ultraclean Diode as Emission Tester for Microwave Application—R. W. Olthuis

Soluble Contaminants

Preparation and Use of High-Purity Intrinsic Water for Electron Device Processing—D. E. Koontz and M. V. Sullivan

The Preparation of High-Purity Methyl Alcohol for Use with Semiconductor Devices—H. F. John

The Utilization of Pure Water in Electronic Device Manufacture—P. P. Pritchett

These papers with discussion were issued as *ASTM Special Technical Publication No. 246*, entitled "Symposium on Cleaning of Electronic-Device Components and Materials."

SUMMARY OF PROCEEDINGS OF THE INTERNATIONAL SYMPOSIUM ON PLASTICS TESTING AND STANDARDIZATION

This International Symposium on Plastics Testing and Standardization was sponsored by ASTM Committee D-20 on Plastics for the American Group of ISO/TC-61. The papers in the Symposium were presented at four sessions. The first session included papers from Belgium, Czechoslovakia, Germany, Hungary, The Netherlands, Poland, Roumania, Sweden, England, the U.S.S.R. and U.S. on "How National Standards Are Achieved." Mr. Frank W. Reinhart, National Bureau of Standards, Chairman of Committee D-20, presided over the session. "Methods of Test for Engineering Properties of Plastics" were discussed in the second session in four papers from France, Sweden, Switzerland, and England under the chairmanship of Mr. Hans W. Paffrath, Farbenfabriken Bayer, Leverkusen, Germany, with Mr. Ralph K. Witt, Johns Hopkins University, as Co-chairman. Four papers in the third session disclosed methods for testing and studying various thermal properties of plastics, the flow test in Germany, variants of the Vicat softening point test in a paper from England, rheological properties of polymers in a paper from the U.S., and the study of impact strength in a French paper. Mr. F. H. Jacobs, Philips, N.V., Eindhoven, Holland, presided over the session as Chairman, with Mr. Paul Willard, Food Machinery and Chemical Corp., as Co-chairman. Four papers from the U.S. in the fourth session discussed methods for molecular characterization of polymers and a paper from Roumania (presented by title only) suggested a classification system for macromolecular products. This last session was presided over by Mr. A. H. Willbourn, Imperial Chemical Industries, Ltd., Hertfordshire, England as Chairman, with Mr. C. H. Adams, Monsanto Chemical Co., as Co-chairman.

Mr. C. A. Webber, E. I. du Pont de Nemours and Co., Inc., was Chairman of the Symposium Program Committee and contributed greatly to the success of the meeting, held in Philadelphia, October 30–31, 1958.

The papers included in the Symposium are:

How National Standards Are Achieved

Introduction—George F. Hussey, Jr.
The Functioning of the Belgian Institute of Standardization and Activity in the Field of Plastics Materials
Czechoslovakian Standards
How German Standards Are Achieved—Gerhard Ehlers
How Hungarian Standards Are Achieved—A Brief Study—Andrew Miklovicz
Standardization in The Netherlands—F. W. R. Wijbrans
Outline of Standardization Problems Relating to Plastics in Poland—Boleslaw Mrozowski

State Office for Standards of the Popular Roumanian Republic
Plastics Standardization in Sweden
British Standards with Particular Reference to Standards for the Plastics Industry—G. Weston and H. M. Glass
The Order of Development and Adoption of Technical Standards for Plastics in the U.S.S.R.
Materials Standards in U.S.A. with Particular Reference to Plastics—Robert Burns
Summary—Robert J. Painter

Methods of Test for Engineering Properties of Plastics

Basic Considerations on the Mechanical Properties of Molded Plastic Objects—P. Dubois, B. Bossu, and M. Chatain
Anisotropic Effects in Testing Plastics—Hilding Hogberg
Encyclopedia of Electrical Insulating Materials—G. de Senarclens
Mechanical Properties of Plastics at High Speeds of Testing—H. Warburton Hall
On the Most Suitable Shape for Plastics Materials Specimens Intended for Measurement of Their Shear Strength—Niuma Goldenberg, Mircea Arcan, and Edward Nicolau (presented by title only).

Thermal Properties of Plastics

Testing Flow Properties of Thermosetting and Thermoplastic Molding Compounds—C. M. von Meysenbug
The Vicat Softening Point Test Method: Correlations, Uses, and Variants—C. E. Stephenson and A. H. Willbourn
Rheology of Polymer Melts—A Correlation of Dynamic and Steady Flow Measurements—E. H. Merz and W. P. Cox
The Function of the Temperature on the Phenomena of Transition in the Study of Impact Strength—Michel Juillard

Methods for Molecular Characterization

The Accurate Determination of Molecular Weights of Macromolecules—Donald McIntyre
The Use of Infrared Spectroscopy in Characterization of Polymer Structure—W. J. Potts
The Use of X-Ray Diffraction and Scattering in Characterization of Polymer Structure—W. O. Statton
Nuclear Resonance Studies of Polymer Chain Flexibility—William P. Slichter
Suggestion for Classification of Macromolecular Products Based on the Correlation Between Structure and Properties of High Polymers—Dan C. Costecu (presented by title only)

The papers with discussion were issued as *ASTM Special Technical Publication No. 247*, entitled "Symposium on Plastics Testing and Standardization."

SUMMARY OF PROCEEDINGS OF THE SYMPOSIUM ON BULK QUANTITY MEASUREMENT

This Symposium on Bulk Quantiy Measurement was sponsored by Research Division II on Measurement and Sampling of ASTM Committee D-2 on Petroleum Products and Lubricants. The installation of automatic tank gages, temperature equipment, and positive displacement meters is becoming widespread throughout the petroleum industry. In many cases, such equipment is being used for custody transfer, and this trend will certainly increase in the years ahead. Eventually ASTM will need to establish standard procedures for the installation, operation, and maintenance of this equipment. The problems associated with standardizing procedures provided a basis for this Symposium, held in Houston, Tex., February 5, 1958, with Mr. E. F. Wagner, The Atlantic Refining Co., acting as Chairman and presiding over the session.

The papers included in the Symposium are:

How Accurate is Hand Gaging?—C. L. Peterson and E. F. Wagner
Automatic Tank Gage Performance—H. E. Sims
Transfers Using Positive Displacement Meters—M. A. Levy
Application, Installation and Maintenance of Automatic Tank Gages—George D. Robinson, Jr.

These papers with discussion were issued as *ASTM Special Technical Publication No. 249*, entitled "Symposium on Bulk Quantity Measurement."

1958 TECHNICAL PAPERS PUBLISHED IN ASTM BULLETIN

ASTM Bulletin *No. 227, January 1958*

The Tower of Babel—A. W. Carpenter, p. 28

Analysis of Radioactivity in Surface Waters—Practical Laboratory Methods—L. R. Setter, G. R. Hagee, and C. P. Straub, p. 35 (TP 1)

Brittleness Temperature Testing of Elastomers and Plastics—A. C. Webber, p. 40 (TP 6)

Mechanical and Physical Characteristics of Jewelry Bronze, 87.5 per cent Strip—Delmar E. Trout, p. 45 (TP 11)

Four Methods of Determination of Carbon Dioxide in Solid Fuels—P. O. Krumin and Karl Svanks, p. 51 (TP 17)

A Laboratory Waxing Machine—F. Walter, R. R. Evans, J. Walker, and R. J. Askevold, p. 58 (TP 24)

The Effect of Loading Rate on Adhesive Strength—Frank Moser and Sandra S. Knoell, p. 60 (TP 26)

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A High-Speed Tension Testing Machine—S. Strella, H. Sigler, M. Chmura, and B. Holman, p. 50 (TP 56)

A Progress Report—Measurement of Surface Moisture—P. J. Sereda, p. 53 (TP 59)

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Criterion for the Evaluation of Roof Coating Asphalts—C. E. Wilkinson, L. Striker, and R. N. Traxler, p. 42 (TP 120)
Nitric-Hydrofluoric Acid Evaluation Test for Type 316L Stainless Steel—Donald Warren, p. 45 (TP 123); Disc., No. 235, January 1959, p. 44 (TP 14)
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A High Shear Rate Capillary Rheometer for Polymer Melts—E. H. Merz and R. E. Colwell, p. 63 (TP 211)

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*A Machine for Evaluation of High-Temperature Alloys Under Combined Static and Dynamic Stresses—Paul E. Hawkes and C. Harold Ek, p. 46 (TP 16); Disc., p. 51 (TP 21)

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* Presented at the Sixty-first Annual Meeting of the Society, June 22–27, 1958.

SPECIAL MEETING FOR AMENDMENT OF ASTM CHARTER*

A special meeting of the American Society for Testing Materials was held at the Headquarters of the Society pursuant to notice to the membership dated October 6, 1958. There were present in person 33 members, being in excess of the quorum prescribed by the By-laws; in addition 3602 members were represented by proxy.

Donald I. Finch, representing Leeds & Northrup Co., nominated Harry M. Hancock, representative attending for The Atlantic Refining Co., as Chairman of the meeting. The nomination was seconded by Mr. F. G. Tatnall, representative for Tatnall Measuring Systems Co.; thereupon a vote was taken and Mr. Hancock was unanimously named Chairman.

Mr. Robert J. Painter, Executive Secretary of the Society, was named Secretary of the meeting.

At the request of the Chairman, the Secretary read the notice to the membership dated October 6, 1958, of this special meeting called for the express purpose of having the membership vote on the proposed charter amendment to revise Section 6 of the Charter, whereby the number of Directors would be increased from fifteen to "eighteen Directors, or such number as shall be prescribed from time to time in the By-laws." The notice was then ordered filed with the minutes of the meeting.

On motion by Mr. Cole and duly seconded by Mr. Thiel, the following resolution was proposed for vote by the membership:

WHEREAS, the Annual Meeting of the Society has voted to refer certain amendments of the By-laws of the Society to a letter ballot of the entire membership in accordance with the terms of Article X of the By-laws; and

WHEREAS, these amendments include, *inter alia*, a proposal to change the number of the Board of Directors; and

WHEREAS, the Charter of the Society specifies the number of the Board of Directors; and

WHEREAS, amendment of Section 6 of the Charter is necessary before the related amendment of the By-laws may become effective; and

WHEREAS, such purpose may be effected by changing Section 6 of the Charter to read as follows:

> "Section 6. The management of the said corporation shall be vested in a Board of Directors, consisting of the President, two Vice-Presidents, the last three Past-Presidents continuing to be members, eighteen Directors, or such number as shall be prescribed from time to time in the By-laws, and such other officers as the corporation may from time to time appoint."

NOW, THEREFORE, BE IT

RESOLVED, that the American Society for Testing Materials, in special meeting assembled, after due and legal notice, in accordance with the laws of the Commonwealth of Pennsylvania, hereby approves the foregoing proposed amendment of Section 6 of the Charter of the Society, as set forth in Resolution adopted by the Board of Directors at its meeting held September 16, 1958, and the President and Executive Secretary are authorized and directed to present to the appropriate court pertinent Articles of

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* Meeting held at ASTM Headquarters, 1916 Race St., Philadelphia 3, Pa., Dec. 17, 1958, at 10:00 a.m.

Amendment to the Charter, and to take such further action as may be required by the laws of the Commonwealth of Pennsylvania to effectuate said amendment.

The Chairman appointed Messrs. W. F. Bartoe, L. P. Mains, and H. W. Stewart as tellers to count the ballots indicated by the proxies and assist in taking the poll of the membership for and against the proposed resolution. The vote was taken on the proposed resolution with the following result: The 33 members present voted unanimously for the adoption of the resolution, and 3570 votes were in favor of the resolution by proxy. Thirty-two negative votes were indicated by the ballots. Accordingly, the Chairman announced that the foregoing resolution had been adopted by the affirmative vote of more than a majority of the members entitled to vote thereon at said meeting of the membership.

The Chairman stated that in accordance with the authority and direction of this resolution, the President and Executive Secretary will proceed to present to the Court, through the Society's counsel, Harry Rosenblum, Esq., Articles of Amendment, and take such further action as may be required by the Commonwealth of Pennsylvania to effectuate said amendment.

The Executive Secretary also announced to the membership that the Society had signed an agreement of sale to purchase premises 1921–23 Arch St. and the properties abutting on the rear, namely, 1920–22 Cherry St., now occupied by The Woman's Christian Temperance Union of Philadelphia, and that settlement would be held January 2, 1959. The membership expressed gratification of this purchase to enable the Society to have extension of its headquarters for the increasing activities of the Society.

Upon motion duly made, seconded and carried, the meeting was adjourned.

ROBERT J. PAINTER,
Secretary.

EDITORIAL NOTE.—The proposed revision of the Charter was approved by the Court under date of February 6, which has been made a matter of record in the Office of the Recorder of Deeds, Philadelphia, as of February 17, 1959.

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1958 LIST OF SPECIAL TECHNICAL PUBLICATIONS

The publications listed below have been compiled during the year 1958 to be issued as separate publications not included in either Proceedings or ASTM Bulletin. For a list of earlier Special Technical Publications, see the original listings in Proceedings, Volume 48, page 1352, and the supplementary listings in Volume 49, page 1240, Volume 50, page 1490, Volume 51, page 1322, Volume 52, page 1309, Volume 53, page 1197, Volume 54, page 1388, Volume 55, page 1250, Volume 56, page 1498, and Volume 57, page 1409. A complete list of Special Technical Publications covering all publications, up to and including 1950, is included in the 50-Year Index to ASTM Publications. Also a complete list up to and including 1955 is published in the 5-Year Index to ASTM Publications (1951–1955).

Special Technical Publication Number	Title
48-G	Alphabetical and Numerical Indexes of X-ray Diffraction Data (1958)
56-L	1957 Supplement to Bibliography and Abstracts on Electrical Contacts (1958)
91-A	1958 Tentative Guide for Fatigue Testing and the Statistical Analysis of Fatigue Data (1958)
227	Thermal Properties of Thirteen Metals—C. F. Lucks and H. W. Deen (1958)
228	Report on Elevated Temperature Properties of Chromium Steels (1958)
229	Symposium on Effect of Ozone on Rubber (1958)
230	Symposium on Elevated Temperature Strain Gages (1958)
231	Symposium on Brittle Failure of Rotor Forgings (1958)
232	Symposium on Soils for Engineering Purposes (Mexico) (1958)
233	Symposium on Radiation Effects on Materials—Vol. 3 (1958)
234	Symposium on Particle Size Measurement (1958)
235	Symposium on Radioactivity in Industrial Water and Industrial Waste Water (1958)
236	Symposium on Some Approaches to Durability in Structures (1958)
237	Symposium on Basic Mechanisms of Fatigue (1958)
238	Symposium on Solvent Extraction in the Analyses of Metals (1958)
239	Symposium on Applications of Soil Testing in Highway Design and Construction (1958)
240	Symposium on Effect of Water on Bituminous Paving Mixtures (1958)
241	Symposium on Paper and Paper Products (1958)
242	Symposium on Bulk Sampling (1958)
243	Symposium on Materials Research Frontiers (1958)
244	Symposium on Stability of Distillate Fuel Oils (1958)
245	Symposium on Advances in Electron Metallography (1958)
246	Symposium on Cleaning Electronic Device Components and Materials (1958)
247	Symposium on Plastics Testing and Standardization (1958)
248	Report on Cast Iron at Elevated Temperatures (1958)
249	Symposium on Bulk Quantity Measurements (1958)
250	Symposium on Instrumentation in Atmospheric Analysis (1958)